VOL. I

No. 8660

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IN THE

United States Court of Appeals

FOR THE FOURTH CIRCUIT

Docket No. 7401

CARL OTTO,
Plaintiff-Appellant,

vs.

KOPPERS COMPANY, INC. and
WHEELING STEEL CORPORATION,
Defendants-Appellees.

APPEAL FROM THE UNITED STATES DISTRICT COURT FOR THE NORTHERN DISTRICT OF WEST VIRGINIA AT WHEELING

JOINT APPENDIX, VOL. I

TESTIMONY—TRIAL AND DEPOSITIONS

W. BROWN MORTON, JR., ESQ.,
ROBERT MCKAY, ESQ.,
PENNIE, EDMONDS, MORTON,
BARROWS and TAYLOR,
New York, New York,

JOHN E. HUBBELL, ESQ.,
MYRON COHEN, ESQ.,
HUBBELL and COHEN,
New York, New York,

FRANK A. O'BRIEN, JR., ESQ.,
O'BRIEN and O'BRIEN,
Wheeling, West Virginia,

For Plaintiff-Appellant.

JO. BAILY BROWN, ESQ.,
KARL B. LUTZ, ESQ.,
BROWN, CRITCHLOW, FLICK
& PECKHAM,
Pittsburgh, Pennsylvania,

WRIGHT HUGUS, ESQ.,
SCHMIDT, HUGUS & LAAS,
Wheeling, West Virginia,

For Defendants-Appellees.

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(7) HAROLD C. COX, having been first duly sworn as a witness, thereupon testified as follows:

Direct Examination by Mr. Morton:

Q. Where do you live, Mr. Cox? A. Johnstown, Pennsylvania.

Q. And what is your present occupation? A. Superintendent of the Coke Plant, Bethlehem Steel Company, at Johnstown.

Q. How long have you held this position? A. Since May 1, 1945.

* * * * *

Q. Now, Mr. Cox, will you tell the Court something about the installation which constitutes the Coke Oven Plant of Bethlehem Steel Company at Johnstown. What sort of plant is this? A. Well, it's the Franklin Coke Plant. It's located in (10) Franklin Borough, which is part of the Johnstown plant of the Bethlehem Steel Company. The Coke Plant at Franklin consists of batteries of coke ovens, and a coal handling division for supplying the batteries with coal, and a coal chemical section for the recovery of the chemical by-products from the gases that come from the batteries.

Q. Does this coal chemical section include apparatus for the recovery of ammonia from the coke oven gas? A. Yes, sir.

Q. Back in 1947 when you wrote the letter [PX 5] to Mr. Hough, about which you have testified a little before, what sort of apparatus was included in the plant, if any, for the recovery of ammonia from the coke oven gas? A. Well, we had four old type cracker pipe saturators that were built, to the best of my knowledge, somewhere around 1921, '22.

Q. What sort of condition were these saturators in in 1947? A. They were in pretty bad condition.

Q. Was it a part of the responsibility of the job that you had as superintendent of the Coke Oven Plant to make recommendations to Bethlehem Company concerning the repair or replacement of these saturators in 1947? A. Yes.

(11) Q. Did you make such recommendations? A. Yes, I did.

Q. What was the recommendation that you made? A. I recommended that the old saturators be replaced with the modern type saturators at that time.

Q. Did you specify in your recommendations any particular type of saturator as desirable? A. Yes, the first recommendation that I made was for an Otto cracker pipe saturator, and later on, why, I changed that recommendation to an Otto spray type saturator.

Q. And as part of your employment and responsibility in that employment, did you visit plants where these various types of saturators were in operation and observe them personally before making your recommendations? A. Yes, I did.

Q. Where did you observe an Otto cracker pipe saturator in operation? A. At the Bethlehem plant, Bethlehem, Pennsylvania.

Q. Where did you observe an Otto spray type saturator in operation? A. At the Lackawanna plant, New York, of the Bethlehem Steel Company.

Q. Am I correct in my understanding that the letter of October 20, 1947, which you wrote Mr. Hough, and of which (12) you have here a copy, you tell us, from the Engineering Department files, is the written record of your recommendations you have just told us about and the reasons for them which you made at that time? A. That's right.

* * * * *

Q. Mr. Cox, when you visited the Lackawanna plant of the Bethlehem Steel Company and observed there an Otto

spray saturator in operation, did you observe its general shape and configuration? A. Yes, sir.

• • • • •

(18) Q. Mr. Cox, you have told us you saw the general shape and configuration of the Lackawanna saturator. Can you tell us whether the saturator as you saw it at Lacka-
(19) wanna was of a different shape from the saturator subsequently installed by you at Johnstown?

• • • • •

A. The Lackawanna saturator was built with a bellied-out section in it so that if it failed as a spray job a cracker pipe could be installed in this bellied-out section, and that is different from the one that we installed at Johnstown, in that the one at Johnstown was made more or less straight up and down, and the sprays installed, with no possibility of ever converting it to this cracker pipe job.

Q. Do you know whether the Lackawanna saturator ever,
(20) in fact, was converted to a cracker pipe job? A. It was not.

Q. In other words, it continued as a spray saturator successfully? A. That's right.

Q. Now, Mr. Cox, you are the Harold Cox, are you not, who executed an affidavit on June 14, 1950, for use in the prosecution of one of Dr. Otto's patent applications? A. Yes.

Q. Have you recently had occasion to re-read that affidavit? A. Yes, I have.

Q. Can you describe to the Court, Mr. Cox, roughly the saturator problem as it existed at Johnstown in 1947, the size of the apparatus, the capacity, and such details as you think would enable the Court to understand the problem that you faced in 1947 with the replacement of saturators, and then describe to the Court your present installation and how it differs? I think probably you can do it better than for me to ask it of you question by question.

A. Well, in around this time, 1945 to 1947, when I became active in the Coke Plant, we had four of the old type cracker pipe saturators. They had been in service (21) since somewhere around 1921 or '22, something like that, and for about ten to thirteen years previous to 1947 they had not been repaired to any great extent, and consequently they were in a very poor condition in 1947. We were having difficulty in keeping them on the line, and the repairs that were necessary were going to be excessive and very expensive, and at that time the Franklin Coke Plant was enlarging its coke producing facilities, so that the load on these old saturators was going to be even greater and they couldn't possibly take care of the current load, so it was certainly to my interest in doing my job and to the interest of the Bethlehem Steel Company to do something about this problem of the old saturators, get them in condition to handle the load properly, and also in condition to handle the increased load, or replace them with something that was more modern and newer. So I knew at that time, of course, in our own company, that our Bethlehem plant was going ahead with some new saturators at their Northampton plant, and I was down and took a look at them, and they certainly did look all right to me. They were an Otto type saturator with cracker pipes, and on coming back from my visit there I recommended to my boss that that looked like it would be the sensible answer to our problem, to replace our four old jobs with a couple of saturators of that type. And along (22) about the same time, of course, why, the job was going in at our Lackawanna plant, and it got into operation, as I remember, sometime about August, 1947, so I made a trip up there to look that job over, and after seeing it in operation and talking to the Bethlehem Steel Company operators, I came back to Johnstown and then I wrote this letter in which I brought out the fact that Lackawanna saturator was operating satisfactorily as a spray type job and it looked to me

as though that was the answer to our immediate problem of taking care of our ammonia removal problem at the Franklin Coke Plant, so that's what I recommended, and stated my reason for recommending it as resulting in power savings and labor savings, and also reduce our losses on sales of sulphate

Q. Was that recommendation acted upon by the company? A. Well, after writing that letter I had several others to write to explain some of the savings, and so forth, to various and sundry people in the Bethlehem organization, but it finally was approved and we got the job installed.

Q. So that now at your Johnstown plant, instead of the four old saturators that were there before this time you have one spray type saturator, is that right? A. That's right.

Q. Am I correct in my understanding from what you have (23) said that the capacity of the one spray type saturator you have there now is greater than the combined capacity of the four that were there before? A. Yes, that's right.

Q. And how about the space occupied by the one spray type saturator compared with the space requirement of the four older ones? A. Well, the Otto spray saturator is occupying the space that was just about occupied by one of the old type saturators.

Q. And is there any difference in the gas pressure drop across the spray saturator as compared to the gas pressure drop across the older type saturators? A. Yes, our spray saturator has a drop that's normally around six to seven inches of water, and the old type saturators, oh, it ran from nineteen inches up to somewhere around thirty-five inches drop.

Q. And it is a fact, is it not, that the lower the drop the less power is required to put the gas through the saturator, is that right? A. Yes, that's right.

(24) Cross Examination by Mr. Brown:

* * * * *

Q. Well now, Mr. Morton brought out that you gave an (25) affidavit on behalf of Otto in connection with his prosecution of a patent on a spray type saturator. You remember that? A. Yes.

Q. Isn't it a fact that that affidavit was prepared by counsel and signed by you, and that you did not yourself write it? A. That's right, yes.

Q. And is it not a fact that you have been somewhat of an advocate of the Otto saturators since, oh, say, 1947, around there? A. Well, I was an advocate for one for myself. I never advocated it to anyone else.

Q. You have been a booster for the Otto saturator, as I understand? A. It has worked very well for us.

Q. Was it your understanding when you gave that affidavit that the use of sprays in connection with saturators was new with Otto?

The Witness: Will you say that again?

Q. Was it your understanding when you gave that affidavit that the use of sprays in connection with taking ammonia out of coke oven gas was something new with Otto as of 1947, or thereabouts? (26) A. Yes, it was my understanding it was new.

Q. So far as you knew, the use of sprays in saturator was new? A. That's right.

Q. Now, I believe you said that these four saturators at Johnstown were in very bad shape and, in fact, as I interpret, were practically to the point where they had to be replaced in 19— A. 47.

Q. (Continuing) —47. Was that when you replaced them? It was later that you replaced them, I believe. A. We replaced them in 1949—

Q. Yes. A. (Continuing)—actually, when we started operation.

Q. And if I understand your testimony correctly, they had just reached the point where they were about falling apart and they had to be replaced, is that right? A. They had to have extensive repairs.

Q. Well now, in this letter that you produced you have given a calculation of an annual labor saving of $32,000.00. I assume that's by putting in the Otto type saturator in place of the four old ones. Do you remember that? A. Yes.

(27) Q. How did you figure that $32,000.00 labor saving? A. Well, with the four saturators we used two men a turn, that's each eight-hour turn. We call them saturator operators. We could eliminate one of those fellows. And then we had dryer operators—

Q. Pardon me. Were there two men for each of the four saturators? A. No, sir.

Q. Two men for the four saturators? A. For the whole thing, yes. And then we had dryer operators—as I remember, we had one on each saturator—and we eliminated those entirely.

Q. Is that elimination of the dryer due to the use of the spray type as distinguished from the cracker pipe type? A. No, not directly. It was due to the incorporation of the automatic sulphate dryer equipment.

Q. Wasn't a good deal of the labor cost of these old saturators due to the fact that they were practically worn out, and they had trouble with the pumps and trouble with piping, and so forth? A. Yes, that's right.

Q. So that there would have been a substantial saving with any new and up-to-date and efficient apparatus put in, I assume? (28) A. No. If the saturators themselves had been in excellent condition, we still would have required those same men to operate them, but the repair

work that was necessary to be done by mechanical means would have been eliminated, of course, for some time, until they wore out again.

Q. Didn't you figure in the mechanical labor of repairs, and so forth, when you were figuring how much you would save by putting in this new saturator? A. No, I was figuring operating saving. I think later on in the letter there you will find the estimated repair expenses that would be eliminated.

Q. Now, if you had put in a new cracker pipe saturator where one installation did the work of the four that were replaced, would you have been able to make the same savings of labor that you have referred to in your letter?

The Witness: Of labor?

Q. Yes. A. Yes, sir.

Q. How do you figure $44,500.00 saving in the cost of power per year due to putting in the one saturator in place of four? A. Well, in order to get gas through the old saturators, it was pumped through by turbines, and approximately (29) forty million cubic feet of gas a day pumped through a saturator bath that's nineteen to thirty inches deep requires more power than what it would take to pump the gas through a saturator with sprays in it where your gas is bubbling through no depth of liquor

Q. But some of your power is required to force the mother liquor through the sprays, I assume? A. That's right, yes.

Q. And the saving in power, as I understand you, is that the difference is due to the head of liquid which you had to overcome in forcing the gas through the liquid, is that right? A. That's right, yes.

Q. And if you had put in a single cracker pipe type installation to take the place of the four, and it had, say, four inches of head over the openings in the cracker pipe,

you would have a substantial saving of power also, is that right? A. Yes. If we had installed the Otto cracker pipe type of saturator the savings in power would have been something over the old saturators, but not as great as the spray saturator job.

Q. Well, putting it in one sentence, the saving of power you refer to is in the amount of power required to (30) force the gas through the saturator, is that right? A. That's right.

Q. I believe you said that due to increased capacity of your by-product coke ovens the necessity for handling more coke oven gas had arisen, is that right? A. That's right.

Q. Is it not a fact that particularly during the war years that all the steel companies were faced with the problem of handling more and more coke oven gas due to increased steel making capacity, and therefore more coke being used in the blast furnaces, is that true? A. Generally, yes.

Q. Now, as far as removal of ammonia from the coke oven gas, the old saturators took the ammonia out, I assume—I mean the old cracker pipe type. A. If they were in good condition they would take the ammonia out.

Q. Unless there was some deficiency, mechanical deficiency, they operated satisfactorily, as far as removal of ammonia was concerned? A. That's right.

Q. And that's true also, is it not, as to the production of ammonium sulphate salt?

The Witness: How was that?

(31) Q. I say, is it not also true that the old cracker pipe saturators when in good condition were satisfactory so far as the production of ammonium sulphate crystals is concerned? A. Yes.

Q. So that, as I understand your testimony, up to this point, the main advantage of the, what you call the Otto type spray saturator, was a saving of power required for

forcing the gas through it, and a saving of space, and of being able to use one instead of four installations, is that about right? A. That's right.

Q. But you haven't said anything, as I recall, about the effectiveness of spray for securing the ultimate result as compared to the cracker pipe in securing the same result. You have talked about cost and space but not as to efficieney in any way. Am I right about that? A. That's right.

(32) Q. As I understand it, the saving of labor due to putting in the Otto spray type saturator as compared to the four old cracker pipe saturators, was that one man could run the new saturator, whereas it required two men to run the four saturators. Is that right? A. Yes, that is right. That is part of the saving. We have dryer operators I mentioned before that were eliminated.

Q. Which was outside of the— A. That was auxiliary equipment to the saturators.

Q. It was an auxiliary, wasn't it? A. Yes.

Q. And they use dryers with the new kind of saturator, do they not, or the equivalent of it? A. That's right.

* * * * *

(39) ROBERT A. BLACKBURN, having been first duly sworn as a witness, testified as follows:

Direct Examination by Mr. Morton:

Q. Where do you live, Mr. Blackburn? A. Ingomar.

Q. What is your present occupation? (40) A. Contract Engineer for Koppers Company.

Q. What generally are the duties of a Contract Engineer for Koppers Company? A. They are to act as a liaison between the customer and the departments of the Koppers Company.

Robert A. Blackburn—For Plaintiff—Direct.

Q. How long have you been employed by the Koppers Company? A. About 20 years.

Q. In what capacities have you worked for the Koppers Company during those 20 years? A. I have worked as an operator in the Research Department, as a Research Engineer. In the Patent Department writing up patent specifications. In the Engineering Department as a Project Engineer, and then later as assistant Chief Project Engineer, as well as Contract Engineer.

Q. Has your employment with the Koppers Company brought you into contact with the construction of apparatus know as ammonia saturators? A. Yes, sir.

Q. Have you ever, during your employment with the Koppers Company, been called on to instruct other Koppers personnel in matters relating to ammonia saturator construction? (41) A. Yes, sir.

Q. What was the nature of that instructional course? A. It was in connection with a course covering coke oven plant design.

Q. Did you prepare the material which constituted that course yourself? A. Yes, sir, with the assistance of the facilities of the company in duplicating and drafting work in connection therewith.

Q. Have you brought with you today a copy of the instructional material relating to ammonia saturators which you so prepared? A. I have, sir.

Q. May I see it? A. It would be lecture 7.

Q. I understand you hand me a book entitled Coke Oven Plant Design, Course K-171, R. A. Blackburn. That is the complete course to which you referred? A. Yes, sir.

Q. And it included as lecture 7, headed Coke Oven Plant Design, By-Product Equipment, Ammonium Sulphate, General, some discussion of the construction and operation of the apparatus we know as ammonia saturators? (42) A. Yes, sir.

Q. The balance of this entire course K-171 was concerned with other aspects of By-Product Plant designs? A. That is true.

Q. I think then I will give you back your book. I understand that the separate lecture 7 you handed me consists of pages 42 through 50, and is the same as what appears in the whole course? A. That's right.

[Said p. marked PX 8.]

Q. Mr. Blackburn, when did you deliver this lecture 7? A. I have given it in four separate years, in the spring of the years, probably, I believe from '49 through '53—1949 through 1953.

(43) Q. Am I correct in my understanding that the notes which have been marked as Plaintiff's Exhibit Number 8 are the notes of the lectures as you gave them in 1949? A. No, sir, they are not.

Q. What year do these particular notes relate to? A. The notes are a development of the material presented during '49, '50 and '51, and actually represent a gradual refining of the material in the course. The last two years I taught the course I furnished them with outlines. Prior to that time they had to take notes. If you are inferring maybe that this is similar material to what we had in 1949, it was not exactly.

Q. These exactly are the outlines which you did furnish the last two years you gave the course. Is that correct? A. That is right, sir.

Q. That would be in 1952? A. I believe that is probably spring of '52 or '53.

Q. Who were the students who attended this course? A. Koppers Company employees.

Q. Where was the course given? A. In the Koppers Building.

Robert A. Blackburn—For Plaintiff—Re-direct.

Q. And the giving of the course was a part of your duties as an employee of the Koppers Company. Is that (44) right? A. No, sir.

Q. Were you paid for it by some other organization than the Koppers Company? A. I was paid for it by the Koppers Company, but it was strictly a voluntary proposition. I did not have to give the course as a duty.

Q. Who was in charge of the course as a whole? A. The training director of Koppers Company.

* * * * *

(50) Re-direct Examination by Mr. Morton:

Q. Mr. Blackburn, would you be able to tell me of your own knowledge how many installations of center dip type saturators the Koppers Company has made and where they are made? A. At Wheeling Steel; at Crucible Steel at Midland, Ohio; at Republic Steel at Warren, Pennsylvania; (51) at Kaiser Company, Fontana, California. That is all that I can recall.

Q. Are any of those installations, as far as you know, still in use? A. As far as I know, two of the installations are still in use.

Q. Which are those two? A. Crucible Steel at Midland, and Republic Steel at Warren.

Q. Can you tell me how many installations of spray type ammonia saturators have been made by the Koppers Company and where they are located? A. I might not be able to give them all, but at Pittsburgh Coke & Chemical, Neville Island.

Q. That is one installation. A. I am starting down the list. At Woodward Steel, Birmingham, Alabama. At Sloss-Sheffield Coke Plant of United States Cast Iron Pipe Company, Birmingham. The Kaiser Steel Company, Fontana. The Wheeling Steel Company, Follansbee. The Republic Steel Company at Youngstown. The United States

Steel and Wire Company at Duluth. The Algoma Steel Company, Sault Ste. Marie, Ontario. That's all I can recall.

Q. Now, Mr. Blackburn, am I correct in understanding that the first such spray installation made was the one made at Neville Island for Pittsburgh Coke and Chemical Company? A. Correct, sir.

Q. Can you tell me whether since that installation was made and successfully operated, Koppers Company has installed any ammonia saturator installation other than of the spray type? (53) A. I don't believe so.

* * * * *

ELLIOTT PRESTON, having been first duly sworn as a witness, testified as follows:

Direct Examination by Mr. Morton:

Q. Where do you live, Mr. Preston? A. Glenshaw, Pennsylvania.

Q. What is your present occupation? A. General superintendent operating department, Koppers Company, engineering and construction division.

Q. What are the duties of that position? A. The overall duties of the position are to see that the coke and chemical plants built by the division go into operation properly.

Q. How long have you held that position with the Koppers Company? A. Since about 1946—'45 or '46.

Q. How long have you been employed by the Koppers Company in any other capacity? A. Since 1924.

Q. What position did you occupy with the Koppers Company in 1941? (54) A. I was assistant operating superintendent.

* * * * *

(63) Q. Now, Mr. Preston, did you have any connection in the course of your employment by the Koppers Company with the construction at Wheeling of the spray type saturators which subsequently replaced the center dip type we have just been talking about? A. It came under the activities of my department to see that it was put into operation, that it got into operation.

Q. Would you be able to say of your own knowledge whether the center dip type installation at Wheeling had worn out or otherwise failed in service prior to the installation of the spray saturators? A. I have no recollection or knowledge of its having worn out?

Q. Now, this morning I asked Mr. Blackburn if he could tell me of his knowledge how many installations of center dip type saturators had been made by the Koppers Company. Could you tell me from your knowledge how many had been made? A. Well, I would find it hard to enumerate them. My (64) recollection is that it was about three or four.

Q. As I recall, specifically, Mr. Blackburn, said that there was an installation for the Wheeling Steel Company at Follansbee, an installation for Crucible Steel at Midland, an installation for Republic Steel at Warren, and an installation for Kaiser at Fontana, California. Can you tell me if there were any others? A. Well, I couldn't identify them at the moment. It's possible, but—

Q. Do you know whether the Koppers Company ever installed a saturator for the American Rolling Mills Company? A. We installed—yes, we installed a spray type saturator for the American Rolling Mills.

Q. A spray type? A. Yes.

Q. Do you recall whether, prior to the installation of the spray type, they installed a center dip type? A. I don't recall it.

Q. Could you recall how many saturators of the spray type the Koppers Company has installed? A. Well, to my

recollection about—one, two, three, four, five, six—my best recollection at the moment would be probably seven or eight.

(65) Q. And does it accord with your recollection, Mr. Preston, that the first spray type saturator installed by the Koppers Company was installed at Neville Island for the Pittsburgh Coke and Chemical Company? A. Yes.

Q. And does it also accord with your recollection, Mr. Preston, that subsequent to the installation of the Neville Island saturator and its operation successfully the Koppers Company has not installed any other than a spray type saturator for any customer in the United States? A. Well, I don't have the chronology of the different units in my mind, but I believe that's substantially correct.

* * * * *

(119) CLYDE C. BIRD, having been first duly sworn as a witness, thereupon testified as follows:

Direct Examination by Mr. Morton:

Q. Where do you live, Mr. Bird? A. Follansbee.

Q. And what is your present occupation? A. By-Product Turn Foreman.

Q. For what company? A. Wheeling Steel Corporation.

Q. And does your job as By-Product Turn Foreman include supervision of apparatus owned by Wheeling Steel Corporation (120) known as the ammonia saturator? A. That's correct.

* * * * *

(128) Cross Examination by Mr. Brown:

Q. Mr. Bird, were you a turn foreman in the operation of the cracker pipe saturators that were at Wheeling be-

fore the present spray type were put in? A. That is correct.

Q. And did you add sulphuric acid to the liquor that was used for removing ammonia in the cracker pipe type of apparatus which was superseded by the present spray type? (129) A. That is correct.

Q. Was the amount of sulphuric acid that you added in the same proportion to the amount of gas that was being treated as in the case of the later spray type saturators? A. Yes, the same amount was used in proportion to the amount of gas we had.

Q. Did you kill, as you have described, in the use of the cracker pipe saturators that preceded the present spray type saturators? A. That is correct.

Q. Did you kill about the same frequency and about the same extent in connection with the cracker pipe as with the present spray type? A. That is correct.

Q. Do the present spray type saturators rest on the same foundations and use the same bowl structure at the bottom of the saturator that the former cracker pipe saturators which were superseded by the present spray type saturators? A. That's correct.

Q. So that, as far as ground space is concerned, the spray type occupy the same space that the two cracker pipe saturators occupied, is that right? A. That's right.

(130) Q. Is there any change in the number of men that operated the two cracker pipe saturators, as distinguished from the present spray type saturators? A. We have added no new personnel.

Q. You use the same men and the same number of men for each of the two types we are referring to? A. That's right.

Q. And of course I am talking about the cracker pipe saturators that were displaced by the present spray type—that's what you understand? A. That's what I understand.

Q. And was the mother liquor, by which I mean the liquor containing sulphuric acid that had passed through the saturator, was that recirculated to the bath in the cracker type saturators? A. Correct.

Q. Now, during the kill operation which you have described and to which Mr. Morton has referred, the passage of coke oven gas through the saturator is not stopped, is it? A. No.

Q. And the gas continues to pass through at the same rate as it does at all other times, as I understand? A. That's right.

(131) Q. And the ammonia is removed even during the killing operation, as I understand? A. That's right.

Q. The only difference is that you don't recover the crystals, but you more or less store them up and take them out at a later time, is that right? A. That's right.

Q. And I believe you said—I may be repeating—that the practice of killing was substantially the same with the cracker pipe as it is with the present spray type? A. That's right.

Q. And for the same purpose generally? A. Same purpose.

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(135) By the Court:

Q. Mr. Bird, you have testified that prior to the installation of the present saturators at Wheeling Steel (136) there were two saturators, have you not? A. That's right, and we have two today.

Q. Yes, and you have replaced those with two that occupy about the same space— A. That's right.

Q. (Continuing) —that the other saturators did. They produce substantially the same result? A. That's right.

Q. In other words, you recover from those ammonium sulphate crystals? A. That's right.

Clyde C. Bird—For Plaintiff—Cross.

Q. Tell me in your own words, and as simply as you can, what the difference is in the process by which the sulphate crystals are removed or recovered.

The Witness: Between the two saturators?
The Court: Yes.

A. Well, the dip type saturator, the gas enters the saturator, it goes down through a pipe which is covered at the end by this bath, and the gas to pass through this bath bubbles up through it, and your ammonia is picked up from the gas by the sulphuric acid in that action. Then as the gas passes on through the saturator, there is a spray, which we call the mother liquor spray, which is used to clean the gas with this high concentrated acid solution, (137) and it passes—

Q. You are still talking about the cracker pipe now? A. That's right.

Q. All right. A. Now, in the spray type saturator, the gas enters and as it first enters the saturator it comes in contact with a spray of mother liquor—or circulated liquor, and that gas then passes around and down through the saturator and comes out and is washed again with the mother liquor high concentrated acid as it leaves the saturator. But the difference is that one saturator has a little differential, which is the spray type saturator, and the other one has a high differential, because there is no bath covering the bottom of the dip pipe in the spray type saturator. Your gas comes in contact with a sprayed mist of liquor.

Q. Your sprayed mist is sprayed from the top of the saturator and sprayed downward, is it? A. That's right.

Q. And that then passes through the gas which is being circulated in that particular chamber? A. That's right.

Q. Then where does the gas go? A. It travels on through, and as it passes out into (138) what we call the

outlet pipe, it is sprayed again by the mother liquor, which you have heard us speak of, high concentrated acid solution, and that cleans it of any excess ammonium that would be in the gas. Then it travels out of the saturator and on to the other equipment.

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(731) CLYDE C. BIRD, who previously testified, was recalled and testified in rebuttal as follows:

Direct Examination by Mr. Morton:

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(733) Q. Now, on the two sheets, Plaintiff's Exhibit 19 and Plaintiff's Exhibit 33, both of these are on the same form, are they not? A. That's right, same form.

Q. There is another heading, "Pressure Inches of Water,"—"In. H_2O," is that right?

(734) And under that is a sub-heading, "Total Diff." What does that tell you about the nature of the data recorded under that heading? A. That's the differential pressure across the saturator itself, gas pressure.

Q. When a change occurs in that total difference or differential across the saturator itself, what does that indicate as to the operation or operating conditions inside the saturator? A. Well, to the operator of the saturator that is an indication of possible salting up of the saturator, or some kind of an obstruction in the saturator, in the gas passage through the saturator.

Q. In the gas passage through the saturator? A. That is right.

Q. Now, just one more thing. If I understand you correctly, or if I understood the nature of this operation correctly, there are sometimes also changes in the spray pressure occurring. What would that indicate, if that pres-

sure fluctuated, to you? A. Well, if the gauge has not been changed, if it's the same gauge we have been using, then I would be under the impression that there would be maybe a build-up of salt in this chamber before our sprays, or possibly in the (735) spray nozzles, or we might have salt building in the pump, the suction of the pump. There's two or three things that could come into that.

Q. Do you also get tar or foreign matter from time to time in the liquor causing clogging of the sprays? A. Not those sprays.

Q. Which sprays might have such a problem? A. We might have a tar build-up of small particles in the mother liquor spray, but in my operation of the saturator I don't believe I have ever found that the circulating liquor sprays ever clogged up due to any foreign matter.

Q. On the print before you there are some sprays marked L and F, which are the main sprays and the mother liquor sprays, is that right? A. The two sprays marked L are the mother liquor sprays, and the ones marked F are the circulating liquor sprays.

Q. Well now, when I talked to you before at Follansbee in January of 1955, I asked you whether you had difficulty with the operation of the saturator through plugging up of the sprays marked on this drawing, Exhibit 4, as L and F, and you said, "Yes, we had, but it was not from salt." A. That is correct.

(736) Q. So that the operation of these sprays has not caused you trouble from salting up but rather from some other form of difficulty? A. That's from other forms of foreign matter that would get in there, such as gaskets or hard particles of tar, or such like. Never have I found it where salt has collected enough to cause us trouble in our sprays.

Cross Examination by Mr. Lutz:

Q. Mr. Bird, referring to this model saturator, which

is Defendants' Exhibit 17, and referring particularly to the sprays in the primary absorption chamber, do I understand you to say that you have never found those sprays clogged up? A. Not with salt.

Q. Have you found any tendency for salt to deposit in those spray nozzles? A. Not in the spray nozzles themselves, no. We have had salt build up a little bit in the chamber in experimenting with our saturator, but not for an ordinary run we have never had any trouble with those sprays salting up.

Q. Mr. Bird, how often do you kill this saturator? A. Our policy has been here the last four to six months of killing it every forty-eight hours.

(737) Q. Would there be any tendency, in your opinion, for those sprays to salt up if you did not kill that frequently? A. No. We have run longer than that and we never had the sprays plug up with salt.

* * * * *

(154) Doctor Carl Otto, having been first duly sworn as a witness, testified as follows:

Direct Examination by Mr. Morton:

Q. Where do you now live, Doctor? A. In Manhassett, Long Island.

Q. You are the Carl Otto who is plaintiff in this proceeding, are you not? A. I am.

Q. What is your present occupation? A. I am the president of the Otto Construction (155) Corporation in New York.

Q. What is the business of that company? A. The building of coke oven by-product plants.

Q. Where were you born, Doctor? A. At Dahlhausen, Germany.

Q. What was your education? A. I went first to Dresden, high school, and then I went to the Institute of Technology at Charlottenburg and from there to Institute of Technology in Breslau. At Charlottenburg I made my Bachelor and Master Degree and at Breslau I got my Doctor Degree.

Q. In what field was your Doctor's Degree? A. It was in Metallurgy.

Q. How long have you been associated with the coke oven industry? A. I was associated with the coke oven industry after I left the Institute of Technology, and even before in my vacations I used to do some practical work on coke oven and by-product plants to familiarize myself with this subject.

Q. What were the dates of the times when you were still in the Institute of Techonolgy? A. I made my Doctor Degree in 1913 and two years before my Master and two years before that my Bachelor (156) Degree.

Q. With what organizations did you obtain practical experience at or about that time in the by-product field? A. I did that with the company of my father in Germany called the Doctor C. Otto and Company.

Q. Is that company still in existence? A. That company is still in existence.

Q. Do you have any connection with it now? A. I have.

Q. What connection? A. I am still the main shareholder of that company.

Q. After you obtained your Doctor's Degree, I believe you said in 1913, did you go to work for that company? A. I did.

Q. In what capacities did you work for that company? A. First as a more or less consulting engineer. I was on the Board and later, from 1925, I was the technical general manager of that company.

Q. What was the business of that company? A. To build coke oven and by-product plants.

Q. Was the business of that company confined to Germany? A. No, it was not confined to Germany. It did (157) business all over the world.

Q. How long did you remain as technical director of that company? A. I remained technical director until 1936 and I was then made Chairman of the Board until 1939 when I left for the U. S. A.

Q. You say you left for the U. S. A. in 1939? A. Yes.

Q. Left from Germany? A. No, from Holland.

Q. When did you leave Germany to go to Holland? A. In 1936.

Q. Did you have, while you were in Holland, any connection with the coke oven industry other than your position as Chairman of the Board of the German company? A. I was then the general manager of the Dutch Company which built coke oven by-product plants in Holland.

Q. What was the name of that company? A. The Otto-Simon Carves Company.

Q. When was the American company of the Otto Construction Company formed? A. That was formed—I could not say correctly—I think either '37 or '38.

(158) Q. It was already in existence when you came to the United States? A. It was already in existence when I came to the United States.

Q. Now I noted in the file history of the center dip patent here in suit, Plaintiff's Exhibit 3, that you are therein described as a citizen of the Empire of Germany, while in the file history of the spray patent in suit, Plaintiff's Exhibit 1, you were described as a citizen of the United States. A. That's right.

Q. I take it that you were naturalized in the period between the filing of the one patent and the filing of the other. Is that right? A. Yes, I was naturalized in November 1943.

Q. What has been the business of the Otto Construction

Corporation in the United States? A. Our business is in the by-product field.

Q. When did you first concern yourself with the apparatus in that field which we have been here discussing, called ammonia saturators? A. Well, I was familiar with ammonia saturators already before, and I saw here the plants to try to sell apparatus to them.

(159) Q. What problems, if any, appeared to you to be confronting the United States coke oven industry with respect to ammonia saturators when you got started over here? A. I found that the conditions in the United States were different as first we had to face here the competition of the biggest by-product coke company in the world, which makes it much more difficult than in Europe.

Q. What was that competition you had to face here? What was the name of your principal competitor as you found it here? A. The main competitor was the Koppers Company.

Q. What other? A. And another company was Wilputte, and he had to face the same competition, and since I have a near friend in Wilputte, he said you do not know what you are against here in the United States. It is different from any other country, and Wilputte also later had to sell out to keep in business to the Allied Chemical and Dye.

Q. Do I understand that you remained the principal owner of the Otto Construction Corporation of the United States? (160) A. That's right.

Q. What other major shareholders are there? A. My son, who is here.

Q. And that is all? A. That is all.

Q. Going back to ammonia saturators, what problems appeared to be confronting the American by-products coke oven industry with respect to their ammonia saturator installations when you were getting started with your Amer-

ican business? A. The problem was that it was the trend of the time, by-product plants were getting too small for the gas output. They wanted to put in more ovens, and because the by-product coke ovens are looked at by the steel plants as a necessary evil, they wanted, of course, to put—to spend as little money as possible for any extension. Therefore, it was important to design apparatus which could take care of this problem and take care of the conditions to get bigger output through the by-product plants, resulting in remodeling the old by-product plant.

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(169) Q. Now you have testified, Doctor, that the apparatus shown on Plaintiff's Exhibit 23 [a flare-mouth cracker-pipe saturator] was actually constructed and installed at Bethlehem Steel plant at Bethlehem, Pennsylvania, and is still there. Can you tell me what individual or individuals you dealt with at the Bethlehem Steel Company in selling them that apparatus? A. You mean the name of the individual I dealt with?

(170) Q. That's right. The names of the individuals, if more than one, that you dealt with at Bethlehem Steel when you sold them that apparatus on that print. A. Mainly with Mr. C. W. Underwood. He was at that time the superintendent of the plant of Bethlehem Steel in Bethlehem.

Q. Was he superintendent of the whole steel plant or coke oven plant? A. He was only the superintendent of the coke oven plant.

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(172) Q. Now, Doctor, let's turn to the other patent here in suit, the patent directed toward the spray type saturators, the patent and the history of its prosecution being shown by Plaintiff's Exhibits 1 and 2. Tell us something about the circumstances under which you were led to the apparatus therein shown. A. The idea of the spray satur-

ator was to improve further the idea of the horizontal flow in the cracker pipe saturator to decrease the pressure difference still further.

Q. When did you first succeed in letting a contract for the construction of apparatus of the spray type? A. We succeeded to get the first contract of this spray saturator by the Bethlehhem Steel Company in August, 1946.

(173) Q. At what plant of Bethlehem Steel Company was this spray apparatus to be installed? A. This apparatus was to be installed at the Lackawanna plant of Bethlehem Steel.

Q. Was the contract for that apparatus as originally let by the Bethlehem Steel for a spray apparatus? A. It was not for a spray apparatus. It was given to us, I think in April or May, for cracker pipe saturator of the same type as built for Bethlehem Steel at Bethlehem.

Q. Can you tell us about the circumstances under which the contract was modified to provide for your spray apparatus? A. It was modified because I had shown my patent application for spray saturator to two gentlemen of the Bethlehem Steel Company.

Q. Who were those two people? A. These two people are Mr. Stahl and Professor Simmons.

Q. By whom was the decision to modify the contract from a cracker pipe, center dip cracker pipe structure to spray considered at Bethlehem, so far as you know? A. I could not mention the person for that. I think that was decided in general in the main office of (174) Bethlehem Steel Corporation, with the local engineers at Lackawanna.

Q. Did you have any dealings with Mr. Underwood in connection with the Lackawanna installation? A. No, I had not.

Q. I now show you Otto Construction Corporation print 2315, dated July 25th, 1946, and I ask you, does that print [See Joint App., Vol. II, p. 347] correctly illustrate the

spray installation for the Lackawanna plant of the Bethlehem Steel Company you have been talking about? A. Yes, it does. It shows the installation which was built. This shows the spray saturator as built at Lackawanna.

Q. Was that the first spray saturator built by you or your company? A. That is the first spray saturator as built by me or the company.

Q. Now is there any point in the gas flow through the apparatus shown on the print before you at which the gas is bubbled through a body of liquid, no matter how shallow? A. No, at no point can the gas bubble through liquor on this drawing.

Q. Was the apparatus which was constructed according (175) to the print before you so built that it could be modified to provide means for bubbling the gas flow through a body of liquid? A. It was. The reason this was provided is—I don't see the dimension—but I think it was twelve feet. The lower part is twelve feet, which would have been the dimension of the cracker pipe saturator in similarity with the cracker pipe saturator at Bethlehem. The upper part decreased then to eight feet, which is in accordance, in coincidence, with the spray saturator if it would have been built without considering that the spray saturator may have failed and would have been remodeled to a cracker pipe saturator.

* * * * *

(176) Q. Dr. Otto, if I recall, before the noon adjournment you had told us certain aspects of the apparatus illustrated (177) on the print (Joint App., Vol. II, p. 347) which showed that it had been installed with possibility that it could be converted back to a center dip cracker pipe type saturator. Was the fact that this structure was so designed known to the Bethlehem Steel Company? A. It was known to the Bethlehem Steel Company that this con-

struction was designed in this way to be changed to a cracker pipe saturator.

Q. And why was it so designed? A. Because Bethlehem Steel didn't feel quite sure that this construction could do what they expected of it.

Q. Could you tell us from recollection, in chronological order, the dates and places at which spray saturator installations built from plans supplied by you or by the Otto Construction Company have been built in the United States? A. The first spray saturator was built at Lackawanna. It was started in August, '47.

Q. By "started," do you mean that the construction was started— A. No, no.

Q. (Continuing)—or the operation was started? A. That the operation was started. I do not remember quite all the dates of the next saturators. I think the (178) next one was built at Johnstown. I couldn't give you the date when it was—when the order was given and when it was started. The following plant was Ford, Detroit. Another plant was in Indianapolis, Citizens Glass. Then Clairton, the first order for three spray saturators. Then Bethlehem Steel, Bethlehem, two spray saturators. And then the last one was three spray saturators for Clairton.

Q. What company operates the plant at Clairton? A. U. S. Steel. The U. S. Steel Company.

Q. And since the installation and successful operation of the spray saturator at Lackawanna, have you or has the Otto Construction Company constructed any plants, any apparatus, for the production of ammonium sulphate from coke oven gas other than of the spray type? A. We have not.

Q. You mentioned that the Bethlehem Steel Company had purchased a spray installation for use at their Bethlehem, Pennsylvania plant. Can you tell me whether that spray installation was used to supplement or whether it

superseded the center dip type installation which you have already told us they had brought for that plant earlier? A. It superseded this installation which they had before of two cracker pipe saturators.

Q. Can you tell us from recollection what the gas (179) through put capacity of the two center dip cracker pipe saturators at the Bethlehem Steel plant was? A. The two cracker pipe saturators had each a through put of fifty-five million cubic feet of gas. We installed, in addition, one saturator to take care of hundred million cubic feet of gas. It was a spray saturator.

Q. That is to say that each of the two cracker pipe saturators could handle fifty-five by themselves? A. Yes.

Q. Or the two together could handle 110? A. That's right, the two together. So these two cracker pipe saturators are a standby for the new spray saturators.

* * * * *

(187) Q. Dr. Otto, it is my understanding from the testimony here today that the Wheeling Steel Corporation always operated these saturators [flare mouth cracker-pipe saturators] installed— A. Singly.

Q. (Continuing)—singly, with the other as a standby. What would have been the effect of putting 74 million— A. Through one.

(188) Q. (Continuing)—through one on the pressure differential at which that saturator operated? A. The pressure difference would have been extremely high then in the newly erected cracker pipe saturator.

Q. And if it had been desired to reduce that pressure differential other than by making a spray type installation, could it have been done by making other changes in the plant? A. Well, that could have been done only—while I do not know the conditions of the other part of the by-product plant, that I assume in this case that they could also have made changes in the pipeline and other apparatus to decrease the load on the gas exhauster.

Carl Otto—For Plaintiff—Cross.

Q. Will you explain to the Court what is a gas exhauster? A. The gas exhauster is the gas pump to pump the gas which is made in the coke oven plant through the by-product plant. It's like a pumping machine.

The Court: I think I understand.

The Witness: And this gas exhauster is provided for a certain quantity, a certain load in pressure. I look at this situation that Wheeling in this case could either put in a saturator like a spray saturator which had the lower (189) pressure difference, or if they would have gone on with their old saturators it would have been necessary for them to make other changes in their by-product plant. I, myself, can, of course, not say here what changes they would have had to make to take this load away from the exhausters, but I think the easiest way to overcome this situation was to put in a spray saturator which had a low pressure difference.

Cross Examination by Mr. Brown:

Q. Doctor, if you took the spray type saturator that you have just been talking about and put through say fifty million cubic feet of gas in a given amount of time, and increased that amount of gas passing through it to say seventy-five million cubic feet, would you not also increase the differential pressure? A. I would.

Q. In other words, if you put more gas through a given saturator as compared to any given time, you will raise the differential that we are talking about? A. Yes.

Q. The differential pressure, or at least part of the pressure that you have been talking about as differential pressure, on the two sides of the saturator in the cracker (190) pipe, is due in large measure to the fact that gas is forced downward in the cracker pipe? A. Right.

Q. And has to overcome the pressure of liquid surrounding the lower end of the pipe, isn't that right? A. That's right.

Q. And the higher the depth, the greater the depth, of liquor surrounding the pipe above the lower outlet end of the pipe, the greater pressure required to force gas through it? A. That's right.

Q. And that's true of all cracker pipe installations? A. That's right, of all cracker pipe installations.

Q. Now, it has been said several times in the testimony, I think in the file history, that the cracker pipe prior art involved heads of liquid of twenty or thirty, maybe more, inches of liquid that had to be overcome by pressure on the gas, is that right? A. That's right. You say twenty-eight inches? A. Twenty-eight?

The Witness: I didn't quite get the figure.

Mr. Brown: I said twenty or more inches.

The Witness: Twenty or more inches?

Mr. Brown: I think there's some reference to maybe (191) more than twenty or thirty.

The Witness: It's twenty, up to thirty, even thirty-five.

Q. Yes, sir, that's what I meant. Now, isn't it a fact that before you made your alleged invention depending on sprays for removing ammonia gas from coke oven gas, that the cracker pipe art had developed cracker pipe installations in which the head of liquor, the depth of liquor above the lower end of the pipe was only three or four inches?

The Witness: In a cracker pipe?

Mr. Brown: Yes.

A. No, I have never—not three or four inches.

Q. Well, how many inches of liquor were there in the cracker pipe installation that you referred to in your direct examination as having been put in at, I believe, Bethlehem? That's what you call the dip type, or something. A. The dip type. At Bethlehem, to my recollection, we had a pressure difference—I talk only of the saturator itself, not of the acid catcher included—of ten inches, and the lowest was about eight inches.

* * * * *

(192) Mr. Brown: Dr. Otto, will you have your counsel give you a copy of [the Otto flare-mouth] patent?

Thereupon, Mr. Morton handed a document to the witness.

Q. Have you it? A. Yes, I have it.

Q. Will you look at Figure 4, Sheet 2, of the drawing? A. Figure 4, yes, sir.

Q. And tell us what the distance a, indicated on the right side of that figure, is, and I am assuming that that distance is the depth of liquor. A. That's right.

(193) Q. Above the lower edge of the cracker pipe, is that right? A. That is correct. That is the depth of the liquor.

Q. Isn't that stated somewhere in the patent to be four inches? A. That is right, but it does not mean that the pressure difference is not only four inches, because, of the horizontal way to the circumference the gas has some additional pressure loss, in addition to the dip in the liquor. You have, of course, to add to the four inches, you have to consider the specific gravity, which is about 1.3, so you have to multiply four by 1.3 to get the water gauge the pressure loss by the dip.

Q. Well, you may be right, to convert a measure of pressure from inches of water to inches of fluid of liquor would require a factor, but my point is not dependent on that. In the cracker pipe described in your second patent in suit, the differential pressure due to forcing the gas through the mother liquor was the force necessary to overcome a head of something like four or six inches, is that right?

The Witness: A head of four, six, inches liquor?

Q. Yes. A. Yes.

(194) Q. And I think that before that, as I understand what you have said in your patent and elsewhere, they had to overcome a head of very much— A. More.

Q. (Continuing)—many more inches of liquor, isn't that right? A. Yes, that's right.

Q. So that at the time of your making your alleged invention of the spray saturator, cracker pipes had also greatly reduced the pressure necessary to force gas through the mother liquor, isn't that right?

The Witness: The cracker pipe lower the dip into the cracker pipe before—do I understand you right?

Mr. Brown: I am afraid I didn't understand your words.

The Witness: I didn't quite get that.

Mr. Brown: Doctor, if you will speak a little slower. We all want to understand you.

The Court: I don't believe he understood your question, Mr. Brown. If you will go back and restate it or have it read to him perhaps we can get an answer.

Thereupon the question was read.

Q. Doctor, by that I mean the depth of insertion of cracker pipes into the liquor bath had been greatly re-

duced in your dip type cracker pipe installation, isn't that, (195) right? A. We reduced the dip in our cracker pipe.

Q. Very materially? A. Yes, very materially.

Q. Doctor Otto, in this cracker dip type, I believe you call it—I have been calling it the bell-mouth type, but you know what I mean— A. I know what you mean.

Q. The construction of your [flare-mouth] patent in suit. When the gas was forced through the cracker pipe and out under the bell-shaped terminal, that gas picked up a lot of liquor and carried it up into the space above the terminal in the form of a spray, did it not? A. Yes.

Q. And that was before your invention of the spray type patent? A. In every cracker pipe saturator when gas bubbles through a liquor something is carried up.

Q. Yes. A. That's right.

Q. But as I understand the theory of your bell-type or flange-type of cracker pipe, you got the gas further away and gave a wider— A. Contact.

(196) (Continuing)—contact, because your gas escaped from a much wider end of the pipe than it did without the bell, is that right? A. That's right.

Q. And now you also called in that patent for the gas going down at great velocity, isn't that right?

The Witness: I didn't get that.

Q. You pumped your gas through this cracker pipe on the [flare-mouth] patent at sufficient velocity to pick up liquor and spray it up through the space above the surface of the liquor bath, did you not? A. Well, I did not increase, especially, the velocity.

Q. You used enough velocity to pick up the liquor and carry it up as a spray into the space above the liquor bath surface? A. Well, but my purpose was really to make it flow horizontally and contact the liquor.

Q. Doesn't your patent talk about picking up the liquor and carrying it up in a geyser effect? A. That is done in every cracker pipe saturator, always parts of the liquor are carried up above the bath.

Q. When you brought, as I understand you, your second patent, the lower edge of the cracker pipe up near the surface of the liquor bath, the gas passing under that (197) surface would pick up more liquor and carry it up into the space above the surface of the bath, would it not? A. I do not think that I had that in mind. I only had in mind to a place, the contact time, of a cracker pipe saturator, which, in the old type, is done vertically, now in a horizontal way.

Q. Regardless of what you were shooting at, will you look at Figure 6 of your second patent in suit? A. Figure 6, yes.

The Court: When you speak of the second patent, do you mean the more recent one?

Mr. Brown: The more recent one, yes, and it is the second patent he put in suit, and what I call the bell-mouth.

The Court: You are talking about—

Mr. Brown: The patent that ends in 794.

The Court: 794, all right. Figure 6?

Mr. Brown: Figure 6, at the bottom of Sheet 2.

Q. Have you that before you, Doctor? A. Yes, I have it before me, Figure 6.

Q. Doesn't that Figure 6 show that the gas passing under the bottom of the cracker pipe and up through the liquor bath picked up, liquor in the form of a spray and took it up into the free space above the bath? A. It shows that the liquor particles are carried away (198) but this same picture you will have in any cracker pipe saturator.

Q. Wouldn't it be more so in the particular one where

the cracker pipe was not immersed as deeply? A. No, I do not think that it is more.

Q. You think it would be the same with any cracker pipe? A. It would be the same with any.

Q. You think you could get that same spray if you had the cracker pipe thirty-five inches below the surface of the bath? A. Yes, because what you mentioned there is new to me. It is quite interesting to get your idea.

Q. Then with all cracker pipe installations the gas passing up through the liquid has carried a spray up into the space above the surface of the liquor, if I understand you correctly, now? A. Yes, to a certain distance, and that is the reason why every cracker pipe saturator, or all saturators, have to use acid catcher, or you can also call it an entrainment catcher.

Q. Dr. Otto, what do you understand to be a spray in the sense of a liquid passed through a gas?

The Witness: You mean what I call a spray?

(199) Q. Yes. What do you mean and what do you understand by the word "spray" in your patent? A. A subdividing of liquor, or a subdividing of a stream of liquor in small particles.

Q. Then if the liquor passed through the gas chamber in small particles it was a spray, is that right? A. That I would not say. I say in the spray you subdivide the liquor in small particles.

Q. Well, would you say that any means that breaks up a stream of liquor into small particles in the gas chamber is a spray? A. No, I wouldn't say that, because a spray is mostly a spray nozzle when you have special means to achieve this.

Q. When you water your lawn with an ordinary garden hose do you call that a spray? A. That's a spray.

Q. When you take a showerbath and have an ordinary head there to divide the water into a stream, do you call that a spray? A. That depends, because you have showerbath with a spray and you have other showerbath, for example, the older ones which are more like a shower that means small openings. I wouldn't call that a spray.

Q. You didn't invent anything yourself in regard to (200) sprays or spray nozzles, so far as your patent on the spray type saturator is concerned, did you? A. No. In connection with the spray type saturators, no, I did not invent a new design of a spray.

Q. As a matter of fact, the sprays that you used and referred to in your patent, and that you use in your saturators, had been on the open market and on sale long before your alleged date of invention in the spray patent, is that right? A. That's right.

Q. And you simply bought them up yourself and put them in your saturator, so to speak? A. That's right.

Q. And you can buy them—they will give you most any kind of spray you want—can't you—coarse sprays, fine sprays? A. That's right.

Q. Streams, mists, atomizers, or what not? A. That's right.

Q. And that was all well long before anything you claim in either patent? A. That's right.

Q. Now, Doctor, I notice in your patent you speak of scrubbers in scrubbing space. What do you understand by (201) "gas scrubber?" A. I understand in the gas scrubber, the space where the gas comes in contact with the liquid where it wants to be contacted with.

Q. For the purpose of taking something out of the gas? A. Yes, for the purpose of taking something out, or for cooling. You can take out something chemically, or you can use for absorption, like you have, for example, scrubbers for the benzol washers.

Q. Well, doesn't the word "scrubbing" imply cleaning or taking something out? A. That's right.

(202) Q. I think in one of your previous answers, Doctor Otto, you said you were familiar with scrubbers. A. That's right.

Q. Before any date we are concerned with here. A. What is that?

Q. Before any date of invention that you claim in your spray patent in suit? A. That's right.

Q. And those scrubbers involved spraying liquids through a chamber containing gas. A. That's right.

Q. And you knew that liquor was sprayed through gas to scrub it, by which I mean taking something out of the gas. Is that right? A. That's right.

Q. Didn't you also know that in connection with ammonia gas, sprays had been employed for bringing liquid into contact with the gas for removing ammonia from coke oven gas? A. Only with packed towers, using water as absorbing liquor, and similar, to take ammonia out of the coke oven gas.

Q. That's all that you knew about the use of (203) spraying something through coke oven gas? A. Taking out ammonia from the gas.

Q. That was the extent of your knowledge, and that was the basis upon which you stated what you believed to be an invention. Is that correct? A. You know from my invention there are several other items which are also included.

Q. I am talking only about the spraying of a liquid such as sulphuric acid solution through coke oven gas. As I understand your answer, you said that you were familiar with spraying water. A. That's right.

Q. Through coke oven gas in packed towers. A. That's right.

Q. And that was all you knew about the spraying of

sulphuric acid solution through coke oven gas? A. Not sulphuric acid solution.

Q. Well, of any solution except water? A. Yes, sir.

Q. That's right? A. That's right.

Q. It was upon that basis that you drew the specifications describing the subject matter which you have patented by this spray patent in suit. Do you (204) understand? A. No, I did not.

Q. Put it this way: You have told us all that you knew about spraying in connection with coke oven gas at the time you filed your application for the patent in suit, 599,067. A. That's right.

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(209) Q. Now, Doctor, the essential feature of the Otto spray saturator, as you understand it, is the spraying of sulphuric acid through an unobstructed space through which coke oven gas passes, isn't it? A. This spraying is not of sulphuric acid. The spraying of an acidified ammonium sulphate solution through an unobstructed space.

Q. Well, as I understand it, you have modified my statement by defining the liquid that is sprayed to be an acidified solution of sulphuric acid containing ammonium sulphate. (210) A. That is right.

Q. Is that right? A. That is right. I changed it.

Q. Now the liquid you are talking about is mother liquor that has previously passed through the saturator and has been drawn off at some place and then is recirculated. Isn't that right? A. That is right.

Q. So that the essential feature then is spraying liquor containing sulphuric acid and ammonium sulphate crystals through coke oven gas. Is that right? A. Not mainly sulphuric acid, but mainly mother liquor containing crystals, this liquor being acidified with sulphuric acid. Sulphuric acid is a small proportion of this liquor.

Q. Well, the liquor that is circulated in your spray type of saturator is simply a part of the mother liquor that has already been used, is it? A. That's right.

Q. Plus the addition of some sulphuric acid? A. That is right.

Q. To make up for what has been lost by ammonium sulphate crystals that have been removed? A. That is right.

(211) Q. And that same kind of mother liquor existed in the cracker pipe saturators, as I understand it. Is that right? A. What?

Q. The same kind of mother liquor also resulted from the use of the cracker pipe saturator? A. It is the same type of mother liquor as in a cracker pipe saturator.

Q. So what I am coming at and getting to, with some circumvention as part of my mistake, sir, is that the real crux of the invention which you are asserting is the spraying of a certain type of liquor through coke oven gas containing ammonia for the purpose of removing the ammonia. Isn't that right? A. That is right.

Q. Now the question I was leading up to, the auxiliary matters—apparatus such as tar catchers, acid catchers, crystal separators, crystal graders, and so forth, is conventional apparatus not peculiar to spray type of saturators at all. Isn't that right? A. That's right.

Q. And such apparatus was old and well known in the art and can be of any conventional type, as I understand it. (212) A. Right.

Q. And can be positioned at any particular place in the installation that is convenient and engineeringly satisfactory. Is that right? A. Right.

Q. And not dependent upon any invention on your part as to where they are placed? A. These additional parts are not part of my construction.

Q. So that what you really want to do by asserting this

067 patent is to prevent everybody else from spraying mother liquor through coke oven gas in an unobstructed space. Isn't that right? A. That is in so far right as we do not want anybody to do this without cooperation with us.

Q. Doctor, some of your claims, and some of your arguments in connection with this 067 patent referred to a whirling spraying. What do you understand by a whirling spray as used in the practice of your invention—your alleged invention? A. A whirling spray in the process of my construction is the spray that is mentioned by name in the application made by me personally, as a Spraco spray.

Q. All you meant by speaking of whirling spray in (213) the argument to the patent office was that you used a Spraco nozzle for producing the spray? A. That is right. But the same principle of this spray you find also in the spray construction of Spraying System in Chicago. It means that the liquor in the spray gets a whirling motion.

Q. While it is in the nozzle? A. Yes, that is right.

Q. And after it gets out of the nozzle, does it move in a spiral path or straight path; that is the individual particles of liquor? A. By the power of energy the drops are separated in small parts by this whirling action.

Q. Isn't it a fact that as soon as they are released from the nozzle and the spiral passage in the nozzle, that each drop is thrown out in a straight line direct, and not the spiral like a corkscrew? A. I certainly think it is not a straight line, because by gravity already you get the power, so under no circumstances is it a straight line.

Q. The individual particle, when it leaves the nozzle, would travel in a straight line except as gravity may tend to draw it towards the earth, or currents of gas might move it sideways, but otherwise it moves (214) in a straight line. Is that right? A. I could not decide that. I think

these special companies, if you are interested in that, they will tell you what these drops do. I know that the drops by this power of energy are separate and in small particles, which is the object of my construction

Q. If you will take a wheel and dip one edge of it in water and whirl it very rapidly it will pick up water and throw it off in the form of drops, won't it? A. I didn't understand that.

Q. If you take a wheel and dip the lower edge of it in water— A. Yes.

Q. —and then rotate the wheel very rapidly, it will pick up liquor— A. Yes.

Q. —and throw it off, or some of it off, in the form of drops. You will admit that, I assume? A. That's right.

Q. And those drops travel in a straight line tangentially to the circumference of the wheel. Don't you know that as an engineer and chemist? A. It will move.

Q. And the tangent—(215) A. Not to the radius.

Q. If they move tangentially they move in a straight line after they leave the wheel? A. Not a straight line, because of gravity, it will be influenced by that.

Q. Yes, of course. A. It is not a straight line.

Q. If you shoot a bullet— A. A bullet also.

Q. Wait a minute. Let me set it up before you knock it down. You said if you have a rifle barrel positioned absolutely horizontal and you fire a bullet out of it, the bullet will start off in a straight line but will be depressed by gravity. A. That is right.

Q. And will have a curve. Is that right? A. That is right.

Q. Now you can't shoot a bullet out of a rifle and make it go in a corkscrew course? A. No.

Q. Can you make it go in a spiral course? A. You can if you give the bullet a special shape.

Q. And that would be caused by friction with the air? (216) A. That's right.

Q. Like a baseball thrown by a good pitcher? A. That's right.

Q. The curve is caused by whirling of the ball in the air? A. That's right.

Q. But except for that, it would go in a straight line, except as gravity or wind changed its direction, wouldn't it? A. That's right.

Q. And the same would be true of drops coming out of a spray, wouldn't it? A. Well, if you disregard—

Q. Disregard gravity and disregard currents of air, it would go in a straight line? A. That's right, it would.

Q. Doctor, it is a fact, isn't it, that the need for handling more and more coke oven gas has grown in the last ten years as the steel industry has grown in capacity, and the demand for steel? A. The need for getting more and more gas?

Q. In other words, the production of coke oven gas is proportional to the production of steel. Isn't that right? (217) A. That's right.

Q. And there has been a growing need for more and more coke ovens, or particularly for coke, and consequently more and more coke oven gas to be handled. Isn't that right? A. That's right.

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(220) Q. You knew Mr. Underwood up at the Bethlehem Steel Company? A. Beg pardon? He is dead.

Q. You did know him? A. Yes.

Q. He was superintendent of the coke oven plant? A. He was superintendent of the coke oven and by-product plant?

Q. And you installed there one of these cracker pipe— A. Two.

(221) Q. —installations? A. Two cracker pipe saturators.

Q. And then later you installed a spray type saturator or saturators? A. That is correct.

Q. Well, now, did you know that Mr. Underwood had himself prior to any time you were at Bethlehem, put spray pipes inside of ammonia saturators at the by-product oven of the Bethlehem Steel Company at Bethlehem? Did you know that? A. No, I did not know that. I heard of this first by the deposition at Bethlehem.

Q. You had never heard of that before? A. I had never heard of it.

Q. You do know now what it was, don't you? A. I know the deposition.

Q. You have seen the drawings of it, haven't you, the drawings of the spray pipes put in by Mr. Underwood? A. No, I don't remember that. I only remember the deposition.

Q. The depositions had with them exhibits which were drawings of the Bethlehem Steel Company showing spray pipes put into the space above the liquor level in the ammonia saturators. (222) A. I did not see these drawings. I only got from Mr. Morton the deposition.

Q. You never even saw— A. The written deposition. I saw that.

Q. You never saw those drawings? A. I didn't see the drawings.

Q. Then I can't very well ask about them— A. No, you can't.

Q. —at this moment. Doctor, the taking of ammonia gas out of coke oven gas is by having sulphuric acid solution brought into contact with the gas containing ammonia. Is that right? A. That is right.

Q. And when brought into contact the reacting takes place in which the sulphuric acid and ammonia combine to form ammonium sulphate. Is that right? A. That's right.

Q. And ammonium sulphate is a solid, a crystal? A. That is right.

Q. And to bring about this reaction and to take the ammonia out of gas, it is desirable to bring the gas into as intimate contact with the liquid containing sulphuric acid— A. That's right.

(223) Q. —as possible? A. That's right.

Q. The better the surface contact of gas with the liquid, the quicker the reaction takes place. Is that right? A. That's right.

Q. Is it not a fact that a spray of dividing liquid into fine particles and forcing it through a gas is the most efficient known means of bringing liquid into contact with a gas? A. It is one means.

Q. Do you know of a better means? A. Well, we have, like in the cracker pipe saturator, you have the bubbling through like the bubbling towers. Your contact can be done in many ways.

Q. My question was, isn't the spray way the most efficient way of bringing the gas and liquid into surface contact, to your knowledge as an engineer and chemist? A. I would say it is one way but I don't say it is the most efficient way.

Q. Well, was it not to your knowledge prior to the time that you claim to have made the invention of this spray type patent, known to you that one way used by (224) chemists was to spray a liquid through a gas where there was to be a reaction or a scrubbing of the gas by the liquor —reaction with or scrubbing by the gas—let me rephrase that.

Didn't you know as an engineer prior to say 1940 that chemists had long used the spray method of bringing a liquid into intimate contact with a gas? A. I knew that sprays were used, but I also knew that in the past all chemists and engineers knew that there had to be done

mostly something additional to get the effect of the real contact, and for this packed towers were used in the past in most scrubbers, and there were many inventions made on the type of filling used in these packed towers.

Q. Didn't you know that benzol had been scrubbed out by spraying a liquid through a gas which was not packed, through gas in a space that was not packed? A. No, that was not known to me. I knew very many different packings used in benzol towers where you wanted a very intimate contact, because the absorption of the benzol is not quite as fast as we would like it to be.

Q. Then do I understand that your knowledge at the time you applied for the 067 spray type patent in suit (225) did not include knowledge that the spraying of a liquid through a gas for the purpose of scrubbing it or creating a reaction between ingredients of the gas and liquid, you didn't know that that process had been used? A. That that was the best process to be used, I did not know.

The Court: He didn't ask you if it was the best. He asked you if you knew those had been used?

A. I knew that sprays were used in open spaces like for example in the cooling main where it was impossible to use packings.

Q. Then you knew that sprays had been used in unobstructed chambers containing gas where it was desired for the liquid to have some effect on the gas? A. That's right, and I knew that this effect was not too efficient.

Q. Do you think the spray type saturator as now made by the Otto Company is more efficient in bringing liquid into contact with ammonia gas than similar use of sprays in unobstructed spaces before your invention?

The Court: Do you understand the question?

A. No, I don't quite see.

The Court: Please read the question.

Thereupon the question was read.

(226) A. I only think it is perhaps more efficient because here we use the contact between ammonia and sulphuric acid. Other sprays in other use have to be designed differently.

Q. Then is there anything about the way you use sprays through an unobstructed space filled with coke oven gas that makes your spray any more efficient than other sprays that have been similarly used for treating gas in an open space? A. You know they also built benzol scrubbers with sprays. They have to be designed differently. So you have to design everything, every apparatus in the right way.

Q. Now, Doctor, do you think that is an answer to my question? Do you seriously think that? A. Because I don't quite get your question.

Mr. Morton: I have some difficulty too.

The Court: Let's have it read. It was not the court's idea that it was responsive.

Thereupon the question was read.

Mr. Morton: If I may inject, I find it difficult to understand exactly what the word "efficient" means in that sentence. If I understand the Doctor's testimony correctly, he has said that the answer depends upon the (227) application—that is, the basic fluids and gases being dealt with. Perhaps you have some more general meaning for the term efficient that would let us get an answer.

Mr. Brown: Well, there is nothing holy about the word efficient. You may take effective, or any synonym you wish.

The Witness: You want—

Carl Otto—For Plaintiff—Cross.

Q. I want to know if you have done something and taught something in your patent that makes the use of the spraying of a solution of sulphuric acid through coke oven gas more effective, more efficient, more valuable or different in any way from prior spraying of sulphuric acid solutions through coke oven gas, and we will prove that that has been done, I think. A. In this general way I could not answer that question.

Q. I don't know whether you mean there is no difference or whether you can't answer the question. I don't know what you mean. A. I can't answer it because you want to compare this with other art such as benzol scrubber in efficiency. I do not know how to do that to my best knowledge.

The Court: That was not my understanding of your (228) question, Mr. Brown. Let's have it read.

Thereupon the question was read as follows: "I want to know if you have done something and taught something in your patent that makes the use of the spraying of a solution of sulphuric acid through coke oven gas more effective, more efficient, more valuable or different in any way from prior spraying of sulphuric acid solutions through coke oven gas, and we will prove that that has been done, I think."

The Court: He is confining his question to the spraying of sulphuric acid solution through coke oven gas in both instances, as I understand.

Mr. Brown: That's right, sir.

A. I would like to say that I do not spray sulphuric acid solution. I spray an acidified solution of saturated ammonium sulphate solution containing crystals through an unobstructed space.

Q. This definition you have given of the acidified solution means simply that it is the mother liquid that is produced in saturators, isn't it? A. That is right.

Q. Is there anything in your patent in the way you (229) do it, or the apparatus used, that gives any different effect to spraying the mother liquid through coke oven gas as distinguished from other cases where mother liquid is sprayed through an open space containing coke oven gas?

Mr. Morton: My objection to that is that this witness has said he knew of no such other case, so if you would define one such other case, I am sure he would make a comparison for you.

Mr. Brown: I think maybe we are getting nowhere because I think the witness—what I was trying to get at was whether or not there was something in the patent, that he had done something that changed the prior art spraying of liquid through a gas in order to produce a reaction between the liquid and the gas for which he could get the benefit. If it does exactly the same thing it always did when you sprayed liquid through gas, then I am going to argue there is nothing to it, just using it the way it had always been used with the same effect. I was giving him a chance, if there is something particular or peculiar about what he does in the way of spraying, I am trying to find out what it is. That is the purpose of my interrogation. I will admit it is hard, and no criticism of Doctor Otto. I have no (230) criticism of him. I think we do have a little trouble with our respective background of languages. He doesn't quite understand me and at times I don't quite understand him, but I am not inferring in the slightest degree any reflection on the Doctor.

The Court: I am not sure whether the Doctor is trying to draw some distinction between the acidified solution of sulphuric acid, which he repeats, and the solution of sulphuric acid. Perhaps that is the gist of the difference between you. I am not sure. I must confess I am confused at some of the answers.

Mr. Brown: The point you make, sir, I tried to cover by asking him if what he meant by the acidified liquor was the mother liquor produced in any—

The Court: And he went through that at some length earlier in his cross examination, and then you seemed to think it was necessary to interrogate him further with reference to that difference. In each instance he says well it isn't the spraying of sulphuric acid through the gas. It is spraying acidified solution of sulphuric acid. Is that it?

Mr. Morton: That's right.

The Court: Is that the correct terminology?

Mr. Morton: The Doctor's last answer was that he (231) was spraying an acidified saturated solution of ammoniam sulphate containing crystals into an unobstructed space.

The Court: Yes. But it is something he gets from the mother liquor some place.

Mr. Morton: That's right, your Honor. It comes right out of the bath at the bottom as shown by the patent drawing, and if I understand and as I recall, when asked about whether that mother liquor was not pretty much the same thing, he said it was. Now the question is whether it was ever sprayed in this manner before. If that is the question.

The Court: I wonder whether we are all together, and I am not sure yet.

Mr. Brown: I am certainly not trying to confuse the Doctor or quibble. I was trying to get it straightened out so we would be all meaning the same thing.

The Court: That's what we are all trying to do, get at the claim of the Doctor as to his invention, and what it covered.

Q. Doctor, you don't add anything to the acidified liquor, as you used the term, in order to produce what is sprayed into the unobstructed space in carrying out your patented process, do you? (232) A. No, I do not add anything besides the replacing always of sulphuric acid in regard to the absorption of ammonia. Nothing is added otherwise to the bath.

Q. In other words, you take out of the mother liquor at some stage a drawoff and you take that up to your sprays and force it down through the gas. A. That is right.

Q. Without the addition or subtraction of anything except perhaps the addition of sulphuric acid to maintain a certain desired strength of solution. A. That is right.

Q. Is that correct, sir? A. That is right.

Q. I will ask one other question. You say ammonium crystals are in the acidified solution that you spray. Those are in there inherently in the mother liquor at the place where you take it off, isn't it? A. Right.

Q. You don't put them in? A. No.

Q. You don't manufacture them for that purpose? A. No.

Q. They are there and they have always been there if you took liquor off at that point in any such (233) arrangement? A. Also the cracker pipe arrangement.

Q. Yes, the cracker pipe or any other type. A. Right.

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(236) Q. Just to avoid confusion, I will say that in refer-(237) ring to your spray type saturator in your examination I am referring to the Wheeling Koppers saturator that is charged to infringe your patent No. 2,569,067. Is that your understanding, Doctor? A. That's my understanding.

Q. And when I have referred or will refer to your cracker pipe type of saturator, I am referring to the structure shown in your patent in suit No. 2,423,794. Is that your understanding? A. That's my understanding.

Q. I am putting this in because Mr. Morton referred to it as a center dip type and I refer to it as a bell-mouth type, but we are talking about these two saturators covered by the two patents in suit, one of which is the spray type and the other of which is the cracker pipe. A. Yes, sir.

Q. Now, Doctor, maybe we can get together on some fundamentals which may be already in the record but scattered through various places, and so I am going to ask you certain questions to see if we can agree on certain fundamentals. As I understand it, the removal of ammonia from coke oven gas— A. That's right.

Q. Now, let me finish my question, please. Maybe (238) you won't agree when I get through. (Continuing question)—the removal of ammonia gas from coke oven gas in the saturators we are talking about is by bringing the gas into contact with the sulphuric acid solution, the sulphuric acid and the ammonia reacting to form ammonium sulphate, is that right? A. That's right.

Q. And I believe it is established by the record that to bring this reaction about it is necessary to bring the coke oven gas into contact with the surface of liquid containing sulphuric acid, is that right? A. That's right.

Q. And I believe we can also agree that the reaction takes place more rapidly and more effectively the greater the contact of the gas with the surface of the liquid containing sulphuric acid, is that right? A. That's right.

Q. And that has been common knowledge long prior to any dates we are concerned with here, is that right? A. That's right.

Q. Can we agree that the greatest surface of a liquid, of a given amount of liquid, with a gas can be produced by dividing the liquid up into small particles, as distinguished from liquid in a basin or pool, is that right? (239) A. I didn't get that.

Mr. Brown: Will you read it, and if you don't understand it please say so and I will try to make it clear.

Thereupon, the question was read.

A. The biggest surface can be achieved by subdividing the liquid in small particles, that's right.

Q. And if you take a given amount of liquid the finer the particle into which it is divided the greater surface contact of liquid there is available for contact with the gas through which the liquid passes, is that right? A. That is right.

Q. In other words, a fine spray of a given amount of liquid gives you a greater surface contact of liquid and gas than a very coarse spray? A. That is right.

Q. And the surface of any liquid divided into drops or particles is greater for a given amount of liquid the finer it is divided, is that right? A. That is right.

Q. In other words, if you took a globule the size of a baseball, assuming that that could be done—which, of course, is not true—and divide it into four, the same (240) amount of liquid, into four spheres, you would have more surface— A. You talk always about the same amount.

Q. Let me finish, please. (Continuing question)— you would have more surface of the liquid than you would have in the liquid in a singly sphere? A. That's right.

Q. Now, all of this was known to chemists and physicists before any date we are concerned with here, I assume? A. It is mathematical.

Q. It's just mathematical, and it's physics, isn't it? A. That's right.

Q. Known to any engineer or physicist or mathematician? A. That is right.

Q. It is a geometrical proposition, in effect? A. No answer.

Q. Two spheres having the same volume as one sphere would have more surface than a single sphere, as I understand it? A. That's right.

Q. Now, Doctor, can we agree that there is no (241) particular virtue to the word "spray" alone when applied to liquid which is exposed in divided form to gas for the purpose of treating the gas with the liquid, either for absorption or for reaction of chemicals in the gas and liquid? If you don't understand the question we will have it read. A. You say was a virtue—

Thereupon, the question was read.

The Witness: I don't understand, a virtue of the spray should be. What do you mean by this.

Q. Well, whether you call it a spray or not, if the liquid is in finely divided form and the gas is passed through a chamber where the divided liquid also passes, it doesn't make any difference whether you call it a spray or call it some other name, does it, as far as your patent is concerned? A. The patent, I think, reads "on sprays."

Q. Well, if you could divide your liquid by any means, whether by spray nozzle or otherwise, so that small particles of the liquid were exposed to the gas, the result would be the same whether you call it a spray or not? That is my point. A. Well, I think in my patent process I only have

used sprays—spray nozzles. I think that makes it more (242) definite.

Q. Can't you answer my question as to whether or not the result would be the same whether you produced the particles by a spray nozzle or by other means, provided the particles of liquid were passed through the gas? A. Well, I think it has been proven already that even spray nozzles give different results, so if you would use other means, which has to be just studied and tested, that that will be the same result.

Q. The ultimate object of any means used to bring gas into contact with liquid in finely divided form is the exposure of the gas to the surface of the liquid, is that right? A. That is right.

Q. And your patent refers to the use of both fine sprays and coarse sprays, and either one can be used to secure the results you aimed at in your patent, is that right? A. That is right, or, I would like to say, as I remember, in my patent I refer to the coarse sprays at the exit to achieve an additional purpose to hit out of the gas the smaller entrained droplet.

Q. In other words, you mean for certain purposes at certain locations you wish a coarse spray and at others (243) you may desire a fine spray? A. That is right.

Q. Now, isn't it also a fact that in the spray coming out of nozzles, such as you describe in your patent, there are globules of liquid of varying sizes? A. That's right.

Q. You can't produce by any means known to you a spray in which all of the globules of liquid are exactly the same size, can you? A. Exactly the same size, I cannot. I would say in every spray the particles always differ in some way. You always have bigger globules and smaller coming out of the same spray.

Q. In other words, it's a kind of random size that comes out of the spray—there are small ones and there are

bigger ones? A. That's right. In my patent I think I also talk about the average size.

Q. By that you mean you would add up the sizes of the little ones and the big ones and divide by two and get an average? A. That's right.

Q. And all of that was known to users of sprays and spray nozzles prior to any date we are concerned with here, 1944 was it not? A. I do not think it was known in connection with our process, because I think otherwise other people would have used it.

Q. Well, Doctor, let's don't argue that. What I want to get out is that physicists, chemists and engineers, knew that nozzles produced varying sizes of globules of liquid that was poured through those nozzles before you made what you call your spray type saturator invention? A. Well, I could say that the people, the companies, who built these spray nozzles, they knew that, that they could deliver to the customers spray nozzles for different globules.

Q. And, as a matter of fact, they advertised before any date we are concerned with here, that they were prepared to furnish nozzles to produce any desired fineness or coarseness of spray of liquid forced through those nozzles, is that right? A. I have not seen that these companies advertised just that they could give different fineness, that when we talked with them that we had this difference in size to face they gave us their advice.

Q. The word "Spraco" has been used. That's a trade name of the spray company which specialized in making spray (245) nozzles, is that right? A. That is right.

Q. That's what we mean when we say Spraco? A. Spraco. It's well known in the United States.

Q. And for the purpose of embodying your alleged invention, you went to the Spraco Company and bought sprays which they had previously been making and selling for producing sprayed liquor forced through the nozzle, is that right? A. That's right.

Q. Now, Doctor, yesterday several times in reply to questions you spoke of using an acidified liquor for spraying into your spray type saturator. Just what do you mean by "acidified?" A. By "acidified liquor," I mean ammonium sulphate liquor saturated, which contains a certain percentage of acid.

Q. And the acid was sulphuric acid? A. Sulphuric acid.

Q. Then isn't it fair to say that when you said "acidified," you meant that the liquor you sprayed there contained sulphuric acid? A. That's right. It contains, for example, between one and a half to ten, or even a few per cent more acid.

(246) Q. And when you took mother liquor from some place in the line of piping or the settlement basins and carried it to the top of your saturator, adding sulphuric acid as needed, you produced an acidified liquor, did you not?

The Witness: What was the beginning?

Thereupon, the question was read.

Q. Let me change the word "produced" to "you had an acidified liquor." A. That's right. I had an acidified. I didn't produce it by pumping it out.

Q. And in the old cracker pipe installations, the mother liquor was an acidified liquor, was it not? A. That was an acidified liquor.

Q. Now, Doctor, your claims and the specification of your spray type patent referred to spraying liquid through ammonia gas in an unobstructed space. What do you understand and what did you mean by the word "unobstructed", as applied to that particular space? A. First, I didn't spray the liquor. You said ammonia through coke oven gas space?

Thereupon, the question was read.

The Witness: You say through ammonia gas?

Carl Otto—For Plaintiff—Cross.

Q. I don't care whether it's ammonia gas or anything else. I am talking about your patent specification and (247) the claims of your patent in suit on the spray type of saturator. A. That referred to spraying a liquid through a coke oven gas containing ammonia, because, excuse me, Mr. Brown, I did not have in mind concentrated ammonia.

Q. I think you are reading into my question something that isn't there. I want to know what you meant by an unobstructed space in your specification and claims. A. By an unobstructed space I mean something contrary to the space as you find in the old type scrubbers which are filled up with some grids or tiles.

Q. In other words, if I understand correctly, and I am asking for information, you were referring to a saturator which did not have grids over which the liquid flowed — A. That's right.

Q. (Continuing)— in order to give surface exposure to the gas? A. That is right.

Q. You referred to taking those out of the chamber and depending upon the spray alone? A. Yes, to have an empty space there.

Q. Well now, would you say that the cracker pipe structure of your cracker pipe patent here in suit (248) embodied an unobstructed space above the bath of mother liquor, and remember that you had a central cracker pipe extended down through the chamber? A. My process refers to spraying liquor into an unobstructed space. A cracker pipe construction is absolutely different from that.

Q. Now, then, will you answer my question? A. You have there not the sprays as I have in my process.

Q. Was there an unobstructed space, in the sense that you use that term in your spray type patent, existing in your cracker pipe installation? A. No, I would not say that.

Q. You would say, then, that if there was a central cylindrical member extending down to a position around

the axis of your spray chamber, that that would be an obstructed chamber for passage of the ammonia gas through it? A. No, I would not, because you can see outside of the cracker pipe the gas goes up and there it goes through an empty space, and you might call that unobstructed.

Q. Did you say obstructed? A. Unobstructed.

Q. Do you not regard the space above the bath of (249) liquor and existing above the liquor bath as it appears in the structure of your cracker pipe patent as being an unobstructed space through which the gas passes? A. Well, I think that's unobstructed.

Q. Now, in your cracker pipe patent, you say that when the gas passes underneath the bell-shaped terminal of the cracker pipe and around the edge and up through the gas, it entrains and carries with it globules of the mother liquor, is that right? A. There is some entrainment carried with the gas. This entrainment does not fill out this unobstructed space to such an extent that it would remove the gas to the expected efficiency. That means, without using the contact under the cracker pipe this apparatus would not give the expected result.

Q. Will you agree that the finely divided liquor carried up into the space which you have said was an unobstructed space— A. Yes.

Q. (Continuing)—in your cracker pipe installation would have a material affect in removing ammonia from the coke oven gas that was passed through the cracker pipe and bath and through the saturator? A. I would not call that a material affect. I would say that that would have some effect in addition to (250) the effect achieved by the contact of the gas with the liquor bubbling through another cracker pipe.

Q. If I understand you correctly, you admit that it would have some effect, but you say that it would not be sufficient

per se, or alone, to take out all the ammonia, is that what you mean? A. That's right, it has some; can be a small effect.

Q. Well, now, you said in a prior answer that the fine particles carried up by the gas did not extend—I forget what you said, but you intimated they didn't extend upward very far—that is, the spray, or whatever you want to call it, did not extend through the chamber, is that right? A. That's right.

Q. Your patent says that the gas passing under the mouthpiece of your cracker pipe carries up mother liquor to wash the walls of the gas treatment chamber and prevent salting thereon. Does that not mean that you considered that the particles carried up by the gas would extend up to and put liquid over all of the walls of the chamber? A. No, it would not, because when we built our saturators we had to install special cleaning sprays at the side to keep the walls clean, so this effect was not really fully achieved as you describe it.

(251) Q. I know it's in your patent. I won't look for it. If your patent says that enough liquor was entrained in the gas in your cracker pipe installation to wash the walls and prevent formation of salt, you now say that is incorrect? A. It is incorrect because we found out that still some cleaning sprays which are supplied with hot water respectively with mother liquor had to be used in addition.

Mr. Brown: I call the Court's attention to the cracker pipe patent, Column 12, beginning with line 20, which says:

"The gas moving rapidly through and out of the emulsified liquor body, B-4, splashes liquor against the gas space wall at a rate appreciably in excess of the rate at which bath liquor adhering to the wall is evaporated by the gas flowing through the gas space."

Q. That statement is correct, is it not? A. I don't remember it that way.

The Court: Let the witness examine it, Mr. Brown, please.

The Witness: That is all right, I believe you, Mr. Brown, that you read it right.

(252) Thereupon, Mr. Brown handed a document to the witness.

The Witness: Mr. Brown, from my knowledge now, I tell you—you want me to—

Q. Yes, go ahead. A. From my knowledge now, I tell you that this does not work in this way as described in this patent, that, in addition, cleaning sprays had to be used.

Q. I believe you testified yesterday that in all cracker pipe saturators, even those in 1916 and 1917, the gas forced down through the cracker pipe and bubbling up through the basin of mother liquor entrained and carried up particles of liquid into the gas space above the surface of the bath, is that right? A. That's right.

Q. I think you said that that was substantially equally true of the old cracker pipe as compared with your patented cracker pipe described in the patent in suit? A. That's right, because I do not think that this patent in suit has more entrainment or works in this respect different from the old type cracker pipes.

Q. Well, will you admit, then, that in all of the cracker pipe installations the passage of gas through the cracker pipe and upward through the liquid picked up (253) particles of liquid and carried them up in the form of small particles into the gas space above the surface of the liquid? A. It carries up some, but not sufficient to take care of the wanted removal of the ammonia from the gas if it could work alone.

Q. But it had a substantial effect in removing ammonia, did it not, this, what I will call spray effect of the gas bubbling through the bath? A. I say not substantial. I say some effect.

Q. This same cracker pipe patent of yours in suit refers to the use of nozzles through the wall of the tank, I suppose you would call it, in which the gas is treated for the purpose of introducing atomized water sprays, is that right? I know it is so.

The Witness: You talk about our spray saturator?

Q. No, I am talking about your cracker pipe. What I am pointing out is that you had nozzles provided in the wall of the tank through which you could introduce water in the form of fine spray. A. That is not right. I had provided pipe lines inside. These pipe lines had holes, and the water sprayed, in effect, out of these holes against the wall.

Q. What kind of nozzles—

(254) Mr. Morton: Mr. Brown, it might help if you gave the witness a copy of the patent which you are asking him to talk about (handing document to the witness).

The Court: Mr. Morton, you have handed the witness a copy of a patent. You might identify it.

Mr. Morton: Copy of the patent, Plaintiff's Exhibit 3, in the case, namely, patent 2,423,794, and unmarked copy.

Q. The only point, Doctor, that I had in mind was to bring out, if it is a fact, that in 1942 you were familiar with the use of spray nozzles for atomizing your liquid. A. I was familiar and knew spray nozzles for atomizing any liquid, but we did not use these in this cracker pipe

saturator for cleaning purpose, because you will see under drawing—I think it was given to you about our cracker pipe saturator for Buffalo—that the pipe lines are provided with holes and not with spray nozzles, because the cleaning of the walls is a different purpose. You would put a cleaning nozzle, for example, also a spray nozzle for water, in an exit pipe to spray against the wall, but to clean the walls in our saturator we wanted to make use of the water in such a way that absolutely most of the water was sprayed against the wall and not in the space of the saturator.

(255) Q. Well, the patent will speak for itself and I don't want to labor the point because, from my point of view, it was a minor thing. I merely wanted to show that you were familiar with the use of atomizing nozzles before any date claimed for your invention of the spray patent in suit. A. In general, certainly I knew atomizing nozzles.

Q. In your deposition, as I recall, you said that when you decided to make a saturator depending upon spraying liquor for removing ammonia gas, one of your problems was to find a non-clogging spray nozzle, is that right? A. That was right, because in my process I had to use these nozzles for a liquor as used in the cracker pipe saturators.

Q. And you solved that problem by going to the spray company and buying one of their conventional commercial nozzles? A. I solved that problem by designing a saturator and installing Spraco nozzles.

Q. Yes. The problem I am talking about was to find a non-clogging spray nozzle. A. That's right.

Q. And you found that already on the market, so that (256) all you had to do was to go out and buy it, is that right? A. Well, that is right, but it gave still some thought because we know now what the tests made for example, at Bethlehem, that nozzles also clog up, and that these tests were given up and that the whole test was a failure.

Q. Well, I think you are trying to load your answer up a little, Doctor, and if you would just answer the question and stop it would please me better. I think it was a simple question and that you answered it in the affirmative, that is, that you solved the problem of finding a non-clogging spray nozzle by buying a commercial nozzle in the open market that had been developed long before any date we are concerned with here. A. Well, I could say it was not—

Q. Can you answer that yes or no? A. No, not in this way. I have to say it was not quite as easy as you put it, because it gave us a lot of thought; otherwise, this would have been done certainly long before by many other engineers.

Q. When you say you gave it a lot of thought, was that uncertainty in your mind as to whether Spraco nozzles would clog or not clog? A. It was—we had to think about would the Spraco nozzles really do the purpose which we expected of them.

(257) Q. And that doubt, if that's what was worrying you, was resolved simply by putting them in and trying them, is that right? A. No, we did not, because these Spraco nozzles we used were too big. We could not just make a test. We designed the saturator for Lackawanna, and we designed it in such a way that in case it would fail we could always go back to the old type of cracker pipe saturator.

Q. As I recall, in your deposition you said that you were uncertain as to whether the Spraco nozzle would work, and you were very much pleased to find when you tried it out in an installation that it did work and did not clog, is that right? A. That's right. We were very pleased that the Lackawanna saturator did not give us any trouble at all.

Q. I feel that it might be pertinent to put it in at this

plage—you had nothing whatever to do with the design of the Spraco nozzles themselves? A. No. The Spraco nozzle is old.

Q. Doctor, correct me if I am wrong, but my understanding is that ammonium sulphate crystals are hard, and you might say sand-like solid, is that right? A. Ammonium sulphate crystals are hard.

Q. Yes. (258) A. Certainly, yes.

Q. So that when you force a stream of liquid containing ammonium sulphate crystals under considerable pressure you have an eroding effect? A. That is right.

Q. Just like a sand blast on the side of a marble wall to clean it, isn't it? A. That is right.

Q. And that was one objection to the use of sprays containing ammonium sulphate crystals with lead-lined saturator types, wasn't it? A. I think you could not build these nozzles and pipe lines out of lead lining, and I don't think they would keep too long.

Q. You think lead would be eroded? A. I think lead would be eroded. We did not try that out.

Q. You didn't have anything to do with investing Monell metal or stainless steel, I presume? A. No, I had nothing to do with it.

Q. You got those on the open market? A. I got those on the open market.

Q. And they are resistant to acid and also resistant to erosion, as compared to lead? (259) A. They are.

Q. Doctor, is it fair to say that even in the cracker pipe art there was a trend towards producing saturators that would have less gas pressure differential on the two sides of the saturator?

The Witness: You mean in the cracker pipe saturator—

Q. Art.

The Witness (Continuing):—art, there was a—well, in that way that everybody tried to keep the dip of the cracker pipe no deeper than necessary.

Q. And, as a matter of fact, there was an evolution in that art producing a decreasing pressure differential over the years?

The Witness: I didn't understand you?

Thereupon, the question was read.

Q. In other words, you yourself have testified that your cracker pipe structure which is described in your cracker pipe patent in suit very materially reduced the differential pressure as compared to certain older cracker pipe installations. That's right, isn't it? A. It reduced materially, but that was due to a different construction of the cracker pipe.

Q. But there was a very material decrease in the differential pressure—(260) A. By our construction—

Q. (Continuing) —in the cracker pipe art before the spray type Otto installation came in? A. I couldn't say that. I only will say that no coke oven by-product builder would use a dip deeper than necessary.

Q. Now, Doctor, that brings up an interesting point.

* * * * *

(267) Q. Doctor, is it fair to say that the crux and (268) essence of what you regard as the invention described and claimed in the spray patent in suit is the use of sprays exclusively to take ammonia out of coke oven gas? A. You talk about the crux and essence?

Mr. Brown: Please read the question.

Thereupon the question was read.

A. Our invention, or our process, is described clearly in the claims of our patent, and that is not as broad as I think you just expressed yourself. It is the spraying of acidified saturated liquor. That means liquor of dissolved ammonium sulphate containing crystals in an unobstructed space.

Q. That is the crux and essence of your invention as you have just stated it? A. That's right.

Q. And that invention, as you understand, would be infringed regardless of the apparatus that was used to practice it, provided the process met the definition you have given. Is that right? A. That is right. That is the process claim. I see here in our claim it is also inferred that this liquor gets supersaturated. It is also mentioned in there.

Q. Let's take one thing at a time. The apparatus (269) for carrying out your invention may be widely varied? A. May be what?

Q. The apparatus for carrying out your invention of your spray patent may be widely varied. Is that correct? A. That is right.

Q. As a matter of fact, your patent shows five different forms on that. A. That is right. We show different forms.

Q. As I understand, two of those have never even been built. Is that right? Figures two and five, as I recall. A. Some have not been built. We have built one in Lackawanna and we have built one in Johnstown, a cylindrical.

Q. You didn't build the apparatus shown in figures two and five, did you? A. In what?

Q. Figures two and five of the drawing of your patent. A. That is right. We have not built them all.

Q. And I think you testified that you had some six or more other pending applications on different forms of apparatus for utilizing sprays. Is that right? (270) Most of which have been abandoned, I believe is what you said.

A. I have not all of that in my knowledge, because I leave this to our patent lawyers. I could not answer that question.

Q. Who is your patent lawyer? A. Mr. Hubbell.

Q. He is the man that wrote all the arguments and amendments to both of the patents in suit, isn't he? A. That is right.

Q. And I believe in your deposition you said sometimes you didn't even see what he said to the Patent Office. Is that right? A. That is right.

Q. And he drew the two affidavits that were filed in the spray case, one of them by Mr. Cox, who testified, and the other by Mr. Pugh. A. That is right.

Q. And Mr. Cox testified that he signed what was sent to him by Mr. Hubbell. A. That is right.

Q. Mr. Pugh entered the employ of Otto some time after he gave an affidavit which was parallel to that (271) of Mr. Cox, did he not? A. That is right.

* * * * *

(278) Q. Doctor, I believe you agree that in use of the word unobstructed space in which mother liquor is sprayed through coke oven gas, the unobstructed space was space without grids or other members over which the (279) liquid would flow. Is that right? A. That is right.

Q. And you knew, did you not, that they had sprayed mother liquor into the top of these packed towers in order to introduce the mother liquor into the treatment chamber? You knew that? A. Yes, I know that from the saturators built by Wilputte for using under-saturated solution in their spray towers which were filled with brick tiles.

Q. So that if you took out the brick, and so forth, out of that saturator and made the spray at the top big enough and used the mother liquor normal to all saturators, you would come out with what you claim is the crux of your invention, would you not? A. No, that is not our in-

vention, because we really changed this Wilputte installation at Lorain. This installation was built with the spray to spray under-saturated ammonium sulphate liquor over tiles. We changed this installation because there was a lot of trouble to this installation. We installed new sprays at the top and we installed additional sprays and used under-saturated liquor. This installation—

Q. You used under-saturated liquor? A. Under-saturated liquor. This installation has (280) nothing to do with what we claim in our patent and is quite a different type of a saturator to take out the ammonia from the coke oven gas. Anybody, any company can use this process.

Q. Provided he puts the packing back in. Is that right? A. No. Our patent is not so broad to cover also that. It is definitely limited to saturated solution and as soon as anybody uses under-saturated solution in the scrubbers and has a separate apparatus where he evaporates this liquor to make the crystals precipitate, he does not infringe our patent.

Q. Referring to that saturated and under-saturated liquor, are you forgetting the patent says you can use for the liquor sprayed into the saturator, saturated, unsaturated liquor? Are you forgetting that? A. That is right, but that means that occasionally the process runs through saturated solution and having even crystals entrained.

Q. We won't get anywhere by arguing that. As I understand, you have no licensees under your spray type patent, have you? A. No.

(281) Q. I understand the Wilputte Company or Allied Chemical have built what you call spray type saturators. Is that right? A. Yes, they have.

Q. Have they taken a license? A. What?

Q. Have they taken a license from you, or asked for one? A. Not yet.

Q. The Spraco nozzle like you have used in all of your spray type saturators is the spray nozzle covered by a certain Eneas patent which you have identified in your file history? A. That is right.

Q. Of spray type apparatus? A. That is right.

Q. Doctor, there might be some little confusion about this, but I think we can agree on it, that for commercial purposes a large ammonium sulphate crystal is preferable. Isn't that right? By large, I mean larger than the fine ones that may be produced. A. That is right. Some people like larger crystals and other people also may think it is not necessary.

Q. And the way of making large crystals instead of (282) fine crystals is by circulation of the sediment of the crystalizing chamber? A. That is right, by running of the plant.

Q. That applies to spray type of cracker pipe type? A. Right.

Q. It is the way you handle the mother liquor? A. That is right.

* * * * *

(283) Re-direct Examination by Mr. Morton:

(291) Q. Dr. Otto, on cross examination you were asked what you meant by whirling spray. I have now obtained and have here a piece of apparatus which I ask you to identify if you can (handing object to the witness). A. This is a spray nozzle made by the Spraying System in Chicago. This spray nozzle has an insert which produces a whirling action inside the nozzle. By this whirling action the centrifugal force begins to act on the globules when they leave the orifice of the nozzle, and by this the liquor stream is divided in small particles outside the nozzle.

Q. I take it that it is the action of a piece of (292) apparatus such as you have shown us here consisting of two parts, the nozzle proper and this insert, which constitute what you have reference to as a whirling spray? A. That's right. The principle is the same as in the Spraco nozzle.

Mr. Morton: I have here and offer in evidence the apparatus which has just been identified by the witness—the nozzle is marked as 26-A and the insert as 26-B.

(Thereupon, the two parts of the Spraying System of Chicago nozzle were received into evidence as Plaintiff's Exhibits Nos. 26-A and 26-B, respectively.)

Q. Now, Dr. Otto, I have here another piece of apparatus. Can you identify that (handing article to witness). A. That's a Spraco nozzle.

Q. By that you mean what? A. That's a nozzle made by another American company. That is Spray Engineering.

Q. The name of the company that makes the Spraco nozzle is Spray Engineering? A. Spray Engineering.

Q. From the point of view of producing a whirling spray, Dr. Otto, is there any difference between the nozzle (293) which has been marked as Plaintiff's Exhibit 26-A and 26-B? A. No, there is no difference in the action. We have used both nozzles side by side, and they give the same effect.

Q. There is an insert similar to the part 26-B inside the Spraco nozzle, is there not? A. Yes, there's an insert, but this you can't take out. This I think is not removable—no, I think it is, but you have to have a screw driver.

Mr. Morton: I might explain I think it is stuck in this one.

Q. Now, Dr. Otto, it might clear up something for the Court—in action, when these nozzles of the type we are talking about are working to produce a whirling spray, does the insert, part 26-B, move or turn within the nozzle, part 26-A? A. No, it should not, because that's the reason why this little set screw is provided.

Q. And it is the reaction of the stream against the insert which causes the stream to turn, is that right? A. That's right.

(294) Q. In your spray saturators, Dr. Otto, do you use spray nozzles made of any particular material for spraying the recirculated bath liquor? A. We like best Alloyco material.

Q. What sort of material is that? A. That's a stainless steel, which is especially hard and stands the erosion perfectly.

Q. Are nozzles made of this material expensive? A. Well, the big nozzles are, and the small ones are less.

Q. For example, a three-inch nozzle, if you know? A. Well, they are more expensive, as the cast iron; they are not too expensive because they amount only to a small part of the whole apparatus.

Q. What would you say, if you know, a three-inch stainless steel spray nozzle of this whirling spray type would cost? A. I couldn't tell you the price, but you get this price from the Spray System Company and the Spray Engineering Company.

Q. Now, Doctor, on your cross examination you were asked just what you considered to be the crux or essence of your invention. Would you tell the Court whether or not it was an object or purpose of your invention merely to (295) provide a means for removing ammonia from coke oven gas? A. No, it was not only the removal of the ammonia, because I think the patent of ours is entitled "Production of Ammonium Sulphate." I think, therefore, that

my process incorporates the removal of ammonia from the coke oven gas and, at the same time, with the same process, the making of ammonium sulphate. I like to compare this with other processes which are used for the removal of ammonia in the coke oven by-product field. These other processes are the removal of ammonia from the coke oven gas by scrubbing the gas with water and making the water absorb the ammonia. To produce, then, ammonium sulphate out of this absorbing liquor, a separate process has to be used in an ammonia still, where the ammonia is driven off from the water which has absorbed ammonia in the ammonia scrubber. So the two processes are here in action. Besides this, there is a third process used for the ammonia recovery. I think I mentioned this this morning by referring to the plant built by the Wilputte Company at Lorain, Ohio. In this plant the ammonia is scrubbed out with undersaturated acidified solution of ammonium sulphate, and the ammonia is absorbed in the liquid by forming ammonium sulphate which is dissolved in the liquid. No crystals are formed. This liquid is then (296) used for a second process where ammonium sulphate crystals are formed in an evaporator. This process consists practically of two processes like the indirect process using water for absorption. The big advantage of our process is to have one process for absorbing the ammonia and for producing ammonium sulphate. Up to now such a process existed in the cracker pipe saturators, but I think that our spray type saturator will replace this construction.

Re-cross Examination by Mr. Brown:

Q. Doctor, so that there may not be any mistake, my understanding is you testified yesterday and testified again today that any whirling there was of the liquid occurred inside of the nozzle? A. That's right.

Q. All before it passed out of the nozzle? A. That's right.

Q. So that there wasn't any whirling of the particles in the sense of projecting them in a spiral fashion after they left the nozzle? A. No.

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R. PHILEMON STOUT, having been first duly sworn as a witness, thereupon testified as follows:

(308) Direct Examination by Mr. Lutz:

Q. Mr. Stout, will you please give us your residence address? A. North Radcliffe Street, Mountwood Route, Bristol, Pennsylvania.

Q. And will you please state your present employment? A. I am assistant chief chemist of the Philadelphia Coke Company.

Q. Mr. Stout, where did you receive your technical education? (309) A. I graduated with a B.S. in Chemical Engineering from the University of Illinois in 1937.

Q. And what was your first employment after you graduated? A. I went to work for Bethlehem Steel Company at Bethlehem, Pennsylvania.

Q. And tell us, briefly, the different positions you have held with that company? A. For the first ten months I was on a training course over the whole steel plant, and then in May, 1938, I went down to the coke plant, where I was continued on a training course in the coke plant for about a year, and then was made an extra shift foreman in the by-product department.

Q. What do you mean by "extra shift foreman"? A. Well, I worked ordinarily on the day turn, but I was available to relieve the regular shift foreman, that is, the men who covered the eight-hour shifts, for vacations, sickness, and did special process work in the by-product department.

Q. Then do I understand that subsequently you became a regular foreman? A. I became a regular turn foreman in February, 1941.

Q. And what section was that in? (310) A. That was in the by-product department.

Q. And this by-product section has supervision over what particular pieces of apparatus? A. That included the primary gas coolers, tar extractors, tar dryers, saturators, ammonia still, light oil scrubbing and recovery plant.

Q. And then I understand that your job as a foreman included supervision over these ammonia saturators, we'll call them? A. On my shift, yes, sir.

Q. And during your duties in supervising these saturators, can you tell us whether or not you became familiar with their construction and operation? A. I did.

Q. Mr. Stout, I show you a drawing which has been marked Defendant's Exhibit No. 1 in this case, which is a Koppers drawing 14,920. Will you please tell us what that drawing shows? A. That shows the original oval cracker pipe saturators as were used at Bethlehem.

Q. Do I understand that's the construction as those saturators were at the time you became a shift foreman? A. Yes.

Q. And you say they were an oval type cracker pipe (311) saturator? A. Yes.

Q. What is the shape of the cracker pipe itself in that saturator? A. In plan view it is U-shaped pipe going around inside of the vessel, at an opening at the bottom with slotted slots to allow the escape of the gas through the liquor.

Q. Mr. Stout, I show you another drawing which has been marked Defendant's Exhibit 6 in this case. It is Koppers drawing No. 22,337. I understand that this drawing shows the general plan arrangement of those old oval saturators, is that correct? A. That is correct, yes.

Q. And how many of these old oval saturators were there in the plant? A. There were five of them there.

Q. And then do I understand that sometime later there were some changes made in these saturators? A. Yes, later spray pipes were added and recirculating pumps were installed outside of the saturators.

Q. And this work was done while you were shift foreman? A. Yes, sir.

Q. And you knew the construction that was being put in (312) at that time? A. Yes, sir.

Q. Did you know the reason for these changes being made? A. Well, one reason was to cut down the differential across the saturator, because they were increasing coke oven capacity, and the need to handle more gas with no greater pressure drop, or possibly less, and it was also expected that the quality of the salt would be improved.

Q. You say the quantity of gas was being increased. What was the reason for this increase in quantity of gas? A. They were putting into operation another battery of coke ovens.

Q. And you say that they expected to improve the quality of the salt. Just what do you mean by that? How was that to be done? A. It was expected by recirculating liquor and crystals that there would be an increase in the size of the crystal from crystal formation from the sprays.

Q. Referring, in general, to these changes which you have mentioned, did you know who originated the idea of these changes? A. As far as I know, Mr. Underwood. We referred to it as "Underwood's Baby."

(313) Q. This was Mr. Charles E. Underwood, as I understand? A. Yes, he was superintendent of the Coke Plant.

Q. What was his position at that time? A. He was superintendent of the Coke Plant, Northampton Division, Bethlehem Steel Company.

Q. Mr. Stout, I show you a drawing which has been marked Defendant's Exhibit 11 in this case. It is Bethlehem Steel Company drawing No. N. 3942. Will you please tell us quite briefly what this drawing shows? A. This is a general arrangement of the saturators, showing the illustration of the spray pipes and the recirculating liquor pumps.

* * * * *

Q. And how many of these circulating pumps were there for each spray saturator? A. There were two pumps for each saturator.

Q. Mr. Stout, I show you another drawing which has been marked Defendants' Exhibit 10 in this case, Bethlehem drawing No. N-3941. Starting at a reference point in the center of this drawing, just above and to the right of center, the red letter J has been applied to a certain part. Will you tell us what that part is, Mr. Stout? A. That's a section of the spray pipe, and it is so identified on the drawing.

(315) Q. In other words, right under it, by the letter G, is the legend "Spray Pipe?" A. That is right.

Q. And what are these things which appear to be projections which stick up and down from that piece of pipe? A. They were deflectors to create a spray from the pipe; that is, at each deflector plate there was a half-inch hole for the discharge of liquor against the spray across this deflector pipe.

Q. Can you tell us from this drawing, Mr. Stout, how far apart these holes were in this spray pipe? A. According to the drawing, there are 4 7/16ths inches between the holes.

Q. Would you infer from this drawing that there was a deflector for each hole in the pipe? A. That is correct.

* * * * *

[Thereupon a chart, DX 14, was discussed. This chart is a simplified drawing based on the two drawings previously discussed.]

Q. Now I wish you would just come down and using this pointer here, tell us a little bit more about the operation. Tell us first, please, Mr. Stout, how the gas passes through this saturator shown on this chart? A. In this elevation view the gas comes in the gas inlet on the left of this view, and passes out through the cracker pipe and passes out through the slots along the bottom of the cracker pipe, and then flows up through the open space in the saturator and out through (318) the gas outlet. In the plan view that would be coming in from the dual inlet and going out on both sides of the cracker pipe, all around the periphery of the cracker pipe in the center of the saturator.

* * * * *

(319) Q. Will you explain how the liquor circulates through that saturator? A. The liquor level is maintained as indicated on the drawing just above the slots. This dual suction pump—the dual pump suctions from that liquor and circulates it back through the U-shaped spray pipe so that it created a spray in the space above the cracker pipe and dropped down into the liquor, so it was maintained in constant circulation.

Q. Am I correct in saying that these red curved lines extending above and below spray pipes represent the path of the spray? A. Yes, as near as I could tell.

Q. And projecting above and below from this spray pipe, I notice a number of little squares. What are they? A. Those squares represent the deflector plates which were welded to the spray pipe at each one of the spray holes.

Q. And you say that liquor was circulated by the pumps up to that spray pipe? A. That is right. There are two

pumps pulling suction from the bath in the saturator, pumping it up. Each pump pumped into one end of this U-shaped loop. It (320) all in one U-shaped pipe.

Q. What would you say the result was of pumping that liquor under pressure through those pipes? What would it do so far as the space within the saturator goes? A. It created a spray which completely covered the cross section, so that the gas in passing up through this open space had to pass through the spray which was being created.

Q. I believe that is all we need on that chart. You may resume the stand. Will you tell me what the composition was of this liquor circulated by the pumps up to the spray pipes? A. It was saturated ammonia liquor, or saturated ammonium sulphate liquor. It was about twenty-five grams per liter sulphuric acid saturated with ammonium sulphate and carried crystals in suspension.

Q. In other words, this was just about the same composition as the liquor that was normally present in an ammonia saturator? A. That is correct.

Q. And the liquor that was present in this saturator after you added the spray pipes, was of substantially the same composition as the liquor present in the old saturators? (321) A. That is right. We didn't change that part of the operation.

Q. Now, Mr. Stout, could you tell us whether or not in the operation of this Underwood saturator using sprays, there were any crystals present in that liquor pumped to the spray pipes? A. Yes, there were.

Q. How could you tell that. A. At one of the points we had a leakage in the packing gland on the pumps, on the circulating pumps, and crystals were present there.

Q. How could you tell that, when it leaked out? A. They were there immediately on leaking out and they also abraded the shaft severely, which was what was giving us trouble with leakage there. There was also evidence in

the saturator. After we had had it in operation for a few months, we developed some leaks of liquor from the saturator, and it was found out these leaks were just below where the spray pipes were around the saturator wall, and the abrasive effect of the crystals had cut the lead lining of the saturator.

Q. Was anything done to repair those leaks? A. Yes. At that time they opened the saturator and built a lead support at the bottom and laid an acid- (322) proof brick lining around the wall of the saturator.

Q. Referring again to that drawing, Defendants' Exhibit 11, that in Bethlehem Steel drawing number 3942, is there any indication on this drawing of that correction you have just said—that change—the adding of the brick lining? A. Yes, in the upper left-hand corner, near the elevation view of the saturator, it shows acid resisting brick four feet high around the side of the saturator, which was added as revision one.

Q. Now you say revision one. Will you please explain to the court where that notation is of revision one, and what it means? A. There is a Roman numeral one which is circled—

Q. Just a minute before you get to the circled number one. I believe it is correct that in this block in the lower right-hand corner of the drawing where the "Bethlehem Steel Company" appears, there is a list of different revisions under the column "revision number." A. Correct.

Q. And a Roman numeral "one" in that column? A. Right.

Q. What does that Roman numeral "one" refer to? A. That refers to this circled Roman numeral one (323) up here at the left middle, which points to this brick lining which was added.

Q. Does that column down there in the name block give you any indication as to the date the change was made?

A. That is dated February 6, 1943, and I think that was installed at just about the same time this drawing was—was placed on the revision—and that was about two or three months after this installation had been put into operation.

Q. In other words then this— A. It was the first difficulty we had had with the operation.

Q. Then the date of this revision number one, when the brick lining was added, is a reference point from which you can state definitely that these saturators with this spray pipe, had been in operation several months prior to this? A. Two or three months at least prior to that date.

Q. That would fix the date of starting at least about the end of 1942. Right? A. That is right.

Q. Now, Mr. Stout, how many of these saturators—I believe you said there were five in the plant, old-style (324) saturators—how many were altered by the addition of spray pipes? A. Two of them were altered originally and placed into operation, and then they went ahead later and altered two more. I think the alteration of the second two was completed after this brick lining was added to the first two. While that was being done we went back to the operation of the other two in the conventional manner.

Q. Can you tell us, Mr. Stout, how much of a job it was to change one of these saturators by adding the pumps and spray pipes? A. Well, it covered several weeks' work by our own construction crew; probably six or eight men, plus a lot longer time by a lead burner in making the necessary revisions to lead lining.

Q. And were any considerable materials involved in such a change? A. Yes, it involved the acid-resisting bronze piping and these deflector plates, and for each saturator two circulating liquor pumps.

Q. Those pumps aren't cheap, are they? A. No, they were not.

Q. Now after these saturators were altered by having (325) the spray pipes, how many of them were operated at one time? A. We normally operated two at a time. In the end there were four converted and we operated two at a time, two running and two spare.

Q. Can you tell us what depth of seal was maintained in these saturators? A. The drawing indicates in the upper left-hand corner a seal of five inches, above the slots.

Q. What do you mean by the seal in this type saturator? A. That is the depth of the liquor above the top of the slots through which the gas escaped from the cracker pipe. We had previously operated with a seal of probably fifteen to twenty inches and the total differential of thirty inches or more. With this installation we were operating with a total differential of fifteen to eighteen inches. That included the acid separators after the saturator.

Q. Now, Mr. Stout, I believe you stated when you were demonstrating on this chart, Defendants' Exhibit 14, that the action of these deflectors was to break the liquor up into fine particles and spray it through a chamber. What was the ultimate effect produced by having (326) this spray present there in that chamber? A. The spray was to remove ammonia from the gas. We reduced the seal so that there was less ammonia being removed in the liquor bath and then created the spray above to remove the balance of ammonia from the gas at reduced over-all differential.

Q. Now, Mr. Stout, do you know of your own knowledge when the Bethlehem Steel Company ceased to operate these Underwood saturators with sprays? A. No, I don't.

Q. What do you know about how long they were operated? A. They were still in operation when I left Bethlehem Steel Company in October 1944.

Q. Now prior to your leaving the employ of the Bethlehem Steel Company in October 1944, had you heard of

any decision to abandon the operation of the spray saturators? A. No, I had not.

Q. I believe you stated previously that these saturators started operating about the end of 1942 and you have just said they were still operating in October 1944. That would be approximately two years. Is that correct? (327) A. That is right.

Q. During this period of approximately two years, how much of the time were these spray saturators operated? A. Except for the period early in 1943 when this brick lining was being installed in the first pair of saturators, they were operated substantially all the time, except for a case of failure of some part of the equipment, which was temporary, because we could always switch over to the two spare saturators.

Q. And during this two-year period, can you tell us about how much gas daily was being passed through these spray saturators?

Q. During most of that period we were producing sixty to seventy million cubic feet of gas a day.

Q. As I understand the purpose of putting in the saturators was to handle an increased volume of gas. A. That is right, to handle an increased volume of gas without increased differential because of the limited gas pumping capacity.

Q. Can you tell us whether the saturators performed the job expected of them? A. Yes, we succeeded in taking care of the gas in a satisfactory manner.

(328) Q. What was done with this gas that was passed through the saturators during this two-year period? A. It was disposed of as underfiring gas on the ovens, as gas in the steel mill and part of it was sold for domestic distribution by the town of Bethlehem Gas Company.

Q. Was that disposition of this gas any different from the way you had disposed of it previous to the installation

of these spray pipes? A. No, that continued to be the same system.

Q. During this two-year period, did you hear any complaints that the gas was of inferior quality? A. No.

Q. Did you know of any difficulties with the salt produced by these saturators? A. No, we had no particular trouble with the salt during this time.

Q. Did you hear of any loss of salt or deficiency in quantity of salt produced? A. None that I knew of.

Q. Were any difficulties encountered in the operation of these spray saturators? A. Well, the biggest difficulty was with the circulating pumps which gave us trouble at the packing (329) gland where we had leakage at the packing gland, and because of the presence of salt crystals and their abrasive and corrosive nature, there was scoring of the shaft and we had considerable difficulty with packing. After the installation of the brick lining, which took up four inches of space between the cracker pipe and the wall of the saturator, so there was smaller space, we experienced more trouble with salting-up of that part of the saturator, and therefore we went to a little stronger acid kill during that time, to keep down the salting-up in the saturators.

Q. Mr. Stout, at this point I wish you would take the pointer again and show the court on the chart, Defendants' Exhibit 14, just where it was that this salting-up occurred that you have been discussing? A. On this section view, section AA, the space between the cracker pipe and the brick wall which was narrower by four inches, probably five inches, than it had been originally. It was close enough that there was a build-up of salt from the liquor—build-up across there.

Q. In other words, the salt had a tendency to bridge over from the cracker pipe to the brick lining. A. That's right.

Q. And it was the presence of this brick lining which (330) narrowed that gap and made it possible for that spot to salt-up? A. It aggravated the condition, yes.

Q. Then as I understand your testimony, all that was needed to overcome this difficulty was that you had to kill a little more frequently than previously. A. That's correct.

Q. Now, Mr. Stout, did you have any trouble with clogging up of these spray nozzles on the spray pipes? A. We had difficulty maintaining circulation because at the far end of the pipe when we were operating two pumps, the velocity was low and the salt would tend to settle out in the pipes, and the pipes would fill up. If only one pump was operating, instead of being the far end, it was back toward the opposite end of the U, and we found several times on opening it up it was the case that the pipe was filled up with salt at the far end rather than—we didn't have any trouble with the nozzles themselves plugging up.

Q. Mr. Stout, I should ask you one other question. I forgot to ask you about this matter of the purple line here. Will you explain what the purple lines are at the top of the plan view and at the left of the section AA? A. Those were the salt slurry pumps which recovered salt and pumped it to the drain tables, or salt dryers.

Q. What was the salt pumped out through these lines? A. It was the ammonium sulphate product.

(332) Cross Examination by Mr. Morton:

Q. Mr. Stout, if I recall correctly, you testified that your knowledge of the path taken by the recirculating bath liquor which passed out of the holes in the pipe marked "spray pipes" on this chart Defendants' Exhibit 14, has been reconstructed by you on the basis of the level given the brick lining later installed. Is that right? A. I did not prepare that drawing.

Q. You did not. How would you, if you were to attempt to reproduce that path which was taken by the material issuing out of the holes in these pipes, how would you, what knowledge would you use, to make such reproduction? A. The bottom discharge could be identified by the point at which we cut through the lining of the saturator which was the upper portion of where the brick was installed, which is about as is shown on that drawing. The spray at the top follows a similar pattern in reverse, and at one time one saturator, when it was open, the pumps were operated with water, to satisfy ourselves that the sprays were covering the entire inside of the saturator.

(333) Q. By covering the entire inside of the saturator, you mean they reached to the top? A. No, not filling the entire space, but clear across the saturator.

Q. Making, in short, a veil or curtain across the cross section of the saturator taken this way (indicating)? A. At least one complete curtain, yes.

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Q. Do you know Mr. Louis A. Farinet? A. Yes, he was one of the other by-product turn foreman.

Q. His job was the same practically as yours on a different shift? A. That's right.

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Q. I believe you testified that one difference between the operation of the saturators with the Underwood modifications and their operation in their unmodified state, was that a more frequent or increased kill of longer duration was required, with the modified saturators than with the un-modified saturators. A. That's right, we used a more severe. I don't think we changed the number of kill. I think we used a more severe kill.

Q. By more severe, what do you mean? A. We killed with acid, where we used 100 grams per liter, we went to about 150 grams per liter.

Q. That would result, would it not, in a longer time when the saturator was not precipitating crystals—would it not. A. That is right.

* * * * *

Q. Now in the course of your examination you had occasion on direct examination once or twice to use the term nozzle. Having reference to the print Defendants' Exhibit 10, which I believe you have with you, Mr. Stout, (342) was there any other structure inserted in the saturators during the time when they embodied the Underwood modifications, any other structure than that illustrated on print 10, which you had in mind when you used the term nozzle? A. No.

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Q. Mr. Stout, do you have any recollection that a structure of the sort shown on the print Defendants' Exhibit 13 I just handed you, was ever installed in the Underwood modification at Bethlehem during your time there? A. I do not.

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Q. I understand you said you were currently employed by the Philadelphia Coke Company as assistant chief chemist. A. Yes, sir.

Q. Does that work involve you in any use of apparatus containing systems for contacting gas with liquid? A. Yes. We run control tests on all of the equipment in the by-product plant, at the coke plant there, and as part of the technical force of the plant are in on most process discussions.

Q. Now I take it from what you have just said, the Philadelphia Coke Company has a coke oven plant of some sort? A. Yes, sir.

Q. Is that a by-product plant or some other form? A. It is a by-product coke oven plant, yes.

Q. And how is the ammonia recovered, or eliminated from the gas produced in that plant? A. In cracker type saturators.

(346) Q. That is a saturator of the same general type in existence at Bethlehem prior to the Underwood modification? A. Basically, except it is a round rather than oval saturator.

Q. Has that saturator ever been modified to include any arrangement similar to the Underwood modification? A. No, it has not.

* * * * *

(348) EDWARD J. HELM, having been first duly sworn, as a witness, thereupon testified as follows:

Direct Examination by Mr. Lutz:

Q. Mr. Helm, will you please give the Court your residence address? A. 475 Sage Drive, Mt. Lebanon, Pennsylvania.

Q. And where did you obtain your technical education, Mr. Helm? A. At the University of Pittsburgh.

Q. What degree? A. Bachelor of Science in Chemical Engineering.

Q. And will you just state, briefly, your experience since graduation? (349) A. I worked for about two and a half years in the Research Department of the M. W. Kellogg Company in Jersey City, New Jersey, working on development of catalytic refining processes. Then came with Koppers Company in January of 1941; with Koppers Company have spent about five years in the Project Engineering Department, about five years in the Operating Department, and approximately five years on development work; for the past three years, as manager of the Development Section of the Engineering and Construction Division of Koppers Company.

Q. Are you a member of any learned societies? A. I am a member of the American Institute of Chemical Engineers; I am a registered Professional Engineer in the

State of Pennsylvania; a member of the Eastern States Blast Furnace and Coke Oven Association.

Q. In connection with your employment with the Koppers Company, have you had occasion to become familiar with the treatment of coke oven gas? A. Yes, I have.

Q. And with the different types of apparatus that are used for that process? A. Yes, sir.

* * * * *

Q. I show you a copy of a chart which has been marked Defendants' Exhibit 15. It is my understanding, Mr. Helm, that this chart which has been marked Defendants' Exhibit 15 was prepared under your supervision, and that it is an exact copy of Figure 3 of this Otto spray patent, is that correct? A. That is correct.

Q. And what, if anything, has been added to this chart No. 1? A. To my knowledge, nothing has been added to the copy (353) which I have. The enlarged chart has been colored to bring out for more—to bring out so that it can be seen more easily the relationship between the gas, liquor and slurry flows.

Q. While you are speaking of the colors, will you just explain to us the colors that are used? Red is used to represent what? A. Red is used to represent the flow path of the liquor.

Q. And blue represents what? A. Blue represents the flow path of the gas.

Q. And what does purple represent? A. Purple represents the flow path of the slurry of crystals removed from the saturator to the feed for the dryer; in other words, that is the path of the slurry product.

Q. Mr. Helm, you said that nothing has been added to this chart, Defendants' Exhibit 15. I believe you meant nothing structurally has been added? A. That is correct.

Q. Actually, the legends have been added, have they not? A. That is correct.

Q. In other words, the names of the different parts (354) have been added in order to help to make that clear? A. That is correct, I beg your pardon.

Q. Mr. Helm, I show you a chart which has been marked Defendants' Exhibit 16. Will you tell us what this chart Defendants' Exhibit 16 shows? A. This chart shows a flow diagram of the Wheeling Steel spray saturator which has been infringed—which has been accused of infringing the Otto patent.

Q. And I believe here, also, this chart, Defendants' Exhibit 16, was prepared by copying exactly a working drawing which is marked, the number of which is marked right on the exhibit, is that correct? A. No, sir, that is not correct. This is a simplified drawing, taken from this 1850-2A10 drawing. The original drawing shows two saturators, one of which normally operates, and the other is a spare. For purposes of simplification, only one of the saturators has been shown on this simplified chart.

Q. In other words, this is a simplified chart prepared from that drawing, but otherwise it is a faithful reproduction of the mechanism shown on that drawing? A. That is correct. To the best of my knowledge, it (355) includes all the essential parts.

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Q. Mr. Helm, I show you a copy of Doherty patent 997,908. Have you studied that patent? A. Yes, sir, I have.

Q. I show you a copy of a chart which has been marked Defendants' Exhibit 19. I believe this chart, Defendants' Exhibit 19, was prepared under your direction from the Doherty patent. Is that correct? A. That is correct.

Q. And I believe the chart shows what should be marked figure 5 in the Doherty patent—

Mr. Lutz: I would like to call the court's attention to the fact that apparently the draftsmen, when

they drew up this chart, failed to put the figure 5 on this figure, and actually figure 5 is at the extreme left side of the sheet of the drawing.

The Court: Well, there appear perhaps to be two figures at the extreme left side; figure 4, the one at the top.

Mr. Lutz: Figure 5 is the one at the lower left corner under figure 4, that's right.

The Court: Yes, sir.

(373) Q. Mr. Helm, referring especially to the chart which has been marked Defendants' Exhibit 19, I would like to ask you a few questions. Does this Doherty patent contain any teaching as to the treatment of ammonia-containing gas with liquor? A. Yes, it does.

Q. Will you tell us the kind of gas which is being treated in this patent? A. It is the gas from coal carbonization, similar in function but different in shape and construction from conventional coke ovens.

Q. And how does the gas itself which is being treated in this Doherty patent compare with ordinary coke oven gas? A. It is very similar and contains ammonia to approximately the same content as coke oven gas.

Q. Does this patent teach anything about the removing of that ammonia? A. Yes, it does.

Q. And tell us briefly how that ammonia is removed according to this patent? A. According to this patent, the ammonia is removed by absorption in a solution containing sulphuric acid, which is sprayed through the absorption section—through (374) the absorption tower.

Q. Will you please take the pointer and explain on the large chart, Defendants' Exhibit 19, just how the gas passes through this absorption tower and how the liquor passes through the tower? First, what is the liquor that is sprayed through there, Mr. Helm? A. The liquor is a

solution of sulphuric acid and ammonium sulphate in water. The gas enters a gas inlet connection near the top of the absorption tower and passes downward through the tower and through the seal device at the bottom of the tower, outward through a gas outlet section near the base of the tower, and up through a gas outlet pipe.

Q. How is the gas indicated on that chart, Defendants' Exhibit 19? A. The flow of gas through the tower is indicated by the blue arrows on the chart.

Q. How about the liquor? How is it circulated? A. The liquor is circulated through the pump marked 64 double prime at the lower left-hand corner of the chart, upward through the connecting pipe to the sprays located near the top of the absorption tower. The liquor is sprayed downward through the unobstructed space in the tower and is collected in the collecting (375) chamber near the bottom of the tower. From this point a portion of the liquor flows back to the recirculating pump 64 double prime for further recirculation to the sprays, while a portion of the liquor flows from the collecting chamber through the pipe 68 prime to the cooling chamber in the adjacent spray chamber, or spray tower, which is marked 65, and is shown as a fragmentary—

Q. Excuse me, Mr. Helm, you said 65. I think you misspoke yourself. A. I mean 25, pardon me.

Q. Where does the liquor go from there? A. The liquor from there goes to the storage tank through the pipe 68 triple prime, the storage tank being marked 70.

Q. What would normally be done with this liquor from this storage tank subsequently? A. This liquor would be, according to the patent as I recall it, processed for crystallization of the ammonium sulphate product.

Q. I don't believe you told us, Mr. Helm, what color this liquor circulation is given on this chart. A. The flow of liquid is shown on the chart by the red marks on the chart.

(376) Q. Does this patent contain any statement as to the fineness of spray? A. Yes, as shown on the chart and quoted from the patent. The patent states "The spray heads produce a very fine spray."

Q. Does the patent say anything about the rapidity of the reaction? A. Yes, again as quoted from the patent on the chart, the patent states, "It is apparent that my method of absorbing the ammonia insures the most intimate contact possible between the gas and the liquid, the absorption of the ammonia being rapid and complete. I thus reduce the capacity necessary in the absorbing apparatus to a minimum."

Q. Now I believe you have said, Mr. Helm, that the reaction in this absorbing tower between the gas and the liquor would produce ammonium sulphate salt, did you not? A. That is correct.

Q. And doing that, would part of the acid be removed from the liquor? A. A part of the acid would be converted chemically to ammonium sulphate, reducing the acidity of the liquor.

Q. And then does the patent say anything about (377) replacing that withdrawn acid with additional sulphuric acid? A. Yes, I believe that the patent states that fresh acid is added as make-up to replace the acid consumed by reaction with the ammonia.

Q. Mr. Helm, I show you a copy of Bosch patent, number 1,029,528. I believe you have studied that patent at our request? A. Yes, sir.

Q. And I show you a copy of a chart which has been marked Defendants' Exhibit No. 20. I believe that chart was prepared under your direction from the Bosch patent. Is that correct? A. That is correct.

Q. Before explaining this chart, Mr. Helm, will you tell us again the system of colors used on this chart? A. Again the blue arrows refer to the flow of gas (378) and the red markings refer to the flow of liquor, or liquid.

Q. Does this patent contain any teaching concerning the use of spray to absorb a gas? A. Yes, it does.

Q. Coming down again and using the pointer, Mr. Helm, will you please explain to the court the flow of gas and liquor in this Bosch patent? A. The chart of the Bosch patent shows apparatus in which the gas enters through the gas inlet connection marked F, near the lower right-hand portion of the apparatus, and flows upwardly through the tower shown at the right-hand portion of the drawing, leaving through a gas outlet connection near the top of the right-hand tower and flowing horizontally through the gas outlet nozzle E and into the gas inlet connection on the left-hand spray tower D. The gas inlet connection to the left-hand absorption tower is near the top of this tower and from this point the gas flows downwardly through the absorption tower D and out through the gas outlet nozzle marked G at the lower left-hand portion of the chart.

Q. How about the circulation of the liquor? A. Liquor is circulated from a collecting chamber marked B through a pump marked A upward through the (379) connecting pipe through a number of spray nozzles marked H, and distributed throughout both of the spray chambers. This liquid is sprayed throughout the entire free space of the two spray towers for the purpose of obtaining intimate contact with the gas.

Q. And what does this patent say as to the fineness of the spray? A. The patent refers to the spray produced by these nozzles as "spray or mist".

Q. And what particular chemical is being removed from the gas in this patent? A. This patent describes apparatus for the removal of oxide of nitrogen.

Q. Does this patent say anything about the size of the apparatus in relation to the amount of gas treated? A. Yes, as shown on the chart and quoted from the patent, the patent states, "In spite of the large quantities of gases

which have to be treated, the apparatus required for the purposes of this invention is comparatively small, and less power is required to keep the gases in circulation."

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Q. Mr. Helm, I hand you a copy of Becker patent No. 1,307,534. I believe you have studied that patent at our request? A. Yes, sir, that is correct.

Q. I hand you a copy of a chart which has been marked Defendants' Exhibit 21. I believe this chart has been prepared under your supervision from that Becker patent. Is that correct? A. Yes, sir, that is correct.

Q. What does this patent show so far as the treatment of coke oven gas is concerned? A. This patent shows the principal items of by-product recovery apparatus required for treatment of coke oven gas.

Q. In other words, we might call it a diagram of (381) the entire process. Is that correct? A. That is correct.

Q. Will you please come down and take the pointer again? And on this large chart, Defendants' Exhibit 21, trace the flow of the coke oven gas and please explain again the colors used on this chart. A. Again the blue lines represent the flow of gas, the red lines represent the flow of liquor, and the purple lines the flow of slurry. With respect to gas flow, gas is shown as it leaves the coke ovens, represented by the figure 5, passes through the gas collecting main which runs along the battery of coke ovens, and flows through the electrical tar precipitator 6. In the tar precipitator the tar fog is removed from the gas. After leaving the precipitator, the gas flows through an acid wash tower 8, where the ammonia is removed by spraying acid solution. After leaving the acid wash tower, the gas flows through a gas cooler 15, where the gas is cooled and a substantial part of the water vapor present in the gas after it had left the collecting mains is removed by condensation. From the cooler the gas flows through

the exhauster 17, which might be considered as a gas pump, and is delivered from the compressor through the saturator 9, in which the gas which has been cooled is (382) contacted with the liquor which has been sprayed into the acid washer tower. In the saturator the gas picks up water, or is saturated with water, and flows from that point through the cooler 20, where the bulk of the water which it has picked up in the saturator is removed by cooling and condensation, and from that point the gas flows through the Benzol scrubber 22, and on to the gas holder, from which point the gas is split into two streams, one being labeled surplus gas, which is available for any desired use, the balance being returned through the line marked fuel gas, for keeping the coke ovens hot.

Q. Part of the gas goes up into this number 25. Did you mention that? A. There may or may not be flow through this line connected to the gas holder. That holder serves to regulate the pressure on the gas line and to take fluctuations in gas production as compared with gas consumption. In other words, this holder has a variable volume which will go up and down, depending upon whether more or less gas is being consumed than is being produced.

Q. May I ask again at this point, I understand in all these charts, the legends have been added, taken (383) from the specifications of the various patents? A. That is correct. These legends in brackets are not shown on the original patent drawing.

Q. Will you please explain the liquor circulation in this patent? A. The liquor circulation with respect to the acid washer tower is shown in red. The liquor is pumped by the pump 13 from the mother liquor tank 12, up to the sprays at the top of the acid washer tower. The ammonia is removed from the gas in the acid washer tower by contact with the acid containing liquor which is sprayed into

the acid washer tower. The liquor which has been sprayed through the gas in this tower is collected at the bottom of the tower and flows directly through the vertical line at the bottom of the acid washer tower into the saturator 9. In the saturator 9 the removal of water from this liquor is obtained by the flow of relatively cool gas after the cooler 95, and the exhauster 17, through the liquor in the saturator, and the removal of water from this liquor by absorption into the gas causes precipitation of the ammonium sulphate in the saturator 9 from the liquor in the saturator 9. Slurry of ammonium sulphate crystals is removed as shown by the purple diagonal line up to the (384) drain table 10 for delivering the crystalline and ammonium sulphate product to the dryers.

Q. I understood you to say, Mr. Helm, that in the saturator 9 the ammonium sulphate crystals are formed? A. That is correct.

Q. Well now, could you state whether or not in your opinion any of those crystals would be present in the liquor circulated to the sprays? A. In my opinion, it is possible with this particular construction that there might or might not be crystals present in the liquor to the sprays, depending upon the conditions of operation.

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Q. Mr. Helm, I show you a photostat of an article from the Hutte Handbook. Will you please tell us first of all in general what this handbook is? A. This Hutte Handbook is a well-known German handbook, this particular copy having been published in 1927, which provides much general information on technical subjects relating to the design of mechanical and processing apparatus and equipment.

Q. Am I right in saying this is a reference book which would be referred to by engineers in Germany? A. That is correct.

Q. I show you a copy of a chart which has been marked Defendants' Exhibit 23. I believe you had this chart prepared from page 220 of the Hutte Handbook. Is that correct? A. That is correct.

Q. Referring to the large chart of Defendants' Exhibit 23, will you please explain the flow of gas (387) and scrubbing liquor through this chart? A. This chart which represents figure 12 from the Hutte Handbook, has been colored to show the flow of gas again with blue arrows, with the gas entering through a connection near the bottom of the spray scrubber and leaving through a connection near the top of the scrubber. The liquid flow is shown by the red lines connected to the sprays in the upper and upper central portion of the scrubber. The flow of slurry, assuming in this case that gas and liquids would be used which would produce a solid material, are shown—the slurry outlet is shown at the bottom of the absorber by the purple line.

Q. I believe this chart contains a section quoted from that handbook. Will you please read that? A. Yes. The chart states, "Spray scrubber without obstructions. Scrubbing liquor is sprayed counter-currently through spray nozzles or the like. Satisfactory for separation of coarse or hygroscopic dusts or for carrying out rapidly proceeding reactions and absorptions. Simple construction, large space requirement, low power consumption, constant pumping output." From page 220 of the Hutte Handbook.

Mr. Lutz: I would like to explain, since this (388) handbook is written in German, the legend which we have copied is a copy of a translation and therefore we did not quote it. It is taken from a translation of the German text, and this has been submitted to our opponents and they have taken no exception to our translation of this passage.

Q. Mr. Helm, I show you a copy of a drawing which has been marked Defendants' Exhibit No. 1 in this case. Would you please tell us what that drawing covers? A. This is a print of a drawing showing the general arrangement of the saturators, or one of the saturators, that was installed around 1917 at the Northampton plant of Bethlehem Steel Company, which is located at Bethlehem, Pennsylvania.

Q. What type of saturator is that? A. We usually refer to this type saturator as the oval shaped cracker pipe saturator.

Q. And I believe, Mr. Helm, I am correct in saying that whenever reference is made to an oval saturator, usually it is that type saturator that is meant? A. That is correct, to the best of my knowledge.

Q. Now I would like to direct your attention to a time about the beginning of 1946. Would you say that (389) that type saturator was a current design in 1946? A. No.

Q. How would you compare it with the designs that were current in 1946, in a general way? A. I would say that this design was obsolete and was incapable of handling nearly so large a gas capacity as the designs which were modern or current in 1946.

Q. Mr. Helm, I show you a copy of Defendants' Exhibit number 11, which is Bethlehem drawing No. X3942. Will you tell us just briefly what is shown on that drawing? A. This drawing is labeled "Northampton plant, saturator house, changes to saturators, general arrangement of piping," and shows in general the changes that were made to install sprays in the old oval cracker pipe saturators at the Northampton plant of Bethlehem Steel Company.

Q. Now I show you a copy, Mr. Helm, of a chart which has been marked Defendants' Exhibit No. 14 in this case. I believe that this chart, Defendants' Exhibit 14, was pre-

pared under your direction from the drawing you have before you. Is that correct? A. That is correct.

Q. And I believe that this chart, Defendants' (390) Exhibit 14, shows accurately, but omitting minor details, the saturator with the spray pipes added as shown on the drawings which you have before you. Is that correct? A. That is correct. That is a simplified chart showing one of the four saturators which were so altered.

Q. Now, Mr. Helm, on the drawing which is Defendants' Exhibit 11, at the bottom of the drawing, just to the left of the center, there is a view called an elevation. Do you follow me? A. Yes, sir.

Q. And on this Defendants' Exhibit 11 a letter M has been applied to a certain structure. Can you tell us what that is? A. Yes, sir, that represents the sprays which were added to the saturator.

Q. In other words, these are what are called "spray pipes" on these drawings? A. That is correct.

Q. And extending above and below the center line of these pipes marked M are certain curved lines. What do those curved lines represent? A. The spray deflectors which were located adjacent (391) to each spray in the spray pipes.

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Q. Then do I understand, Mr. Helm, that these curved (392) parts which you have referred to as deflector plates would break the stream of liquid up into streams? A. Yes, sir. Well, I would say that they would break the streams of liquid up into droplets.

Q. Have you had any previous experience, practical experience, with similar types of plates in breaking a stream up into droplets or spray? A. Yes, Koppers Company has for many years used what we call splash type, or splash plate type sprays or spray nozzles, which are so designed that the liquid being sprayed flows through an orifice in

the pipe in a solid stream and this stream impinges on a deflector or splash plate which is mounted several inches below the bottom of the orifice and held in that position by spider-like supports. In many instances, there are two of these deflector plates suspended one below the other and both below the bottom of the spray orifice. Usually a smaller orifice is located in each of the lower deflector plates. The purpose of this design is to permit the solid stream of liquid flowing through the nozzle orifice to impinge upon the deflector plates and to splash therefrom and be broken up into many droplets, individual spray droplets.

Q. Based on your experience with that type of spray (393) nozzle which you have referred to as a splash plate spray, would you give us your opinion as to whether or not these deflector plates illustrated on the Underwood saturator, Defendants' Exhibit 14, would be effective in breaking that liquor sprayed under pressure into relatively fine streams? A. In my opinion that would be effective, and I would say that they would break the liquid up, not particularly into streams, but rather into individual particles or droplets.

Q. Now, Mr. Helm, you have referred to these splash plate sprays with which you have had some experience. Will you tell us briefly what the advantages and disadvantages of that type spray are? You mentioned some of the advantages. Just summarize them briefly. A. The advantages of a splash type plate spray are (394) that relatively they are designed or fabricated with the largest possible passages which would tend to make them the type most free from tendency to clog, as compared with other nozzles, such as the Spraco or Spraying system type nozzle.

Q. How about ease of cleaning? A. When or if they are used with systems that result in clogging, in spite of the relative freedom from clogging tendencies, they can

easily be cleaned by rodding—sticking a rod through the nozzle and connecting piping without shutting down the apparatus or withdrawing the nozzle and spray pipes from the apparatus.

Q. Are there any disadvantages to this type spray? A. Yes, I would say that the principal disadvantage is the fact that this type spray does not provide as uniform a distribution of the spray particles as does a so-called Spraco or full cone type spray.

Q. You make reference to the Spraco nozzle. Will you just briefly summarize the advantages and disadvantages of the Spraco nozzle? A. As compared with the splash plate nozzle, I think the advantages and disadvantages are practically reversed. The Spraco type nozzle has relatively smaller (395) passages, speaking of the minimum restrictions in each type nozzle, which would be the most likely spots for clogging to occur. On the other hand, the Spraco type nozzle definitely provides a more uniform spray which provides a cone of spray in which the droplets are quite uniformly dispersed.

Q. Then, Mr. Helm, considering the fact that each of these types of spray nozzles has its advantages and disadvantages, how would you, as an engineer, decide which of these types to use in a particular location? A. I would, I believe, make such a decision entirely upon the basis of the likelihood of the particular system involved to result in clogging of the sprays, considered in combination with the need to obtain extremely uniform distribution of the spray throughout the area into which it is desired to disperse the spray. Those two considerations would have to be weighed and in many cases, they are opposing considerations. And it would have to be decided which of the two limitations was more important.

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Q. Mr. Helm, I show you a copy of Jeremiassen patent 2,375,922. I believe you have studied this patent at our request? A. Yes, sir.

Q. And I show you a copy of a chart which has been marked Defendants' Exhibit No. 24. I believe this chart was prepared under your supervision? A. That is correct.

Q. And I believe this chart corresponds with Figure 2 (398) of the Jeremiassen patent? A. That is correct.

Q. Does this patent contain any teaching concerning the absorption of ammonia from gas? A. Yes, it does.

Q. Will you please take the pointer there and explain to the Court the showing on Figure 2 of the Jeremiassen patent. A. Figure 2 of the Jeremiassen patent shows an apparatus for the contacting of gas with a shower of liquid drops for the purpose of absorbing a material such as ammonia from the gas. Shall I describe the flow?

Q. Yes, and I understand that in one particular instance described in the patent the gas does contain ammonia? A. That is described in the patent.

Q. And in that particular instance what sort of liquor is circulated? A. Ammonium sulphate liquor—saturated ammonium sulphate liquor containing sulphuric acid.

Q. And here again the red indicates liquor and the blue indicates gas? A. That is correct.

Q Will you please now describe the flow of gas and (399) liquor through this apparatus? A. The gas enters through the gas inlet nozzle 5, shown at the left of the drawing, and passes inwardly and is distributed around the periphery of the gas inlet chamber by the baffle arrangement, which is designated by the number 20. The gas then flows inwardly radially toward the center of the contacting chamber, flowing through the shower of liquid droplets. The gas then flows upwardly to the gas outlet located at the top of the apparatus.

Q. Now, how does the liquor circulate? A. The liquor is

circulated by the pump, designated with the figure 1, through the pipe connected to the perforated distribution apparatus located near the top of the gas contacting space. The liquor is then distributed in the form of a shower through the perforated pipe inside the gas contacting space, and falls down in what is described in the patent, as I recall, as a rain of drops, down through the gas, and is collected in the collecting space beneath the gas contacting chamber.

The liquor then flows from that point by gravity through the U-shaped pipe connected to the bottom of the crystallizing chamber 11, and flows from that point upward through the suspension of ammonium sulphate crystals to the liquor outlet connection near the top of the crystallizing (400) chamber which connects to the pump.

Q. And what is the nature of that liquor that is circulated? How would you describe the liquor? A. The liquor is a saturated solution of ammonium sulphate containing free sulphuric acid and ammonium sulphate crystals.

Q. In that respect, how does that liquor correspond with the liquor that is circulated through the Wheeling saturator? A. It would be exactly the same type of liquor.

By the Court:

Q. Referring to Exhibit No. 24, I see the legend, "Supersaturated Solution," and then quoting, "Within the Metastable Limit." A. Yes, sir.

Q. Will you explain what that means? A. I will try. I might say, your Honor, that the concept of metastable supersaturation, in my opinion, is a little difficult to describe, but my understanding, as applied to this apparatus and process, is that a solution (401) which is supersaturated within the metastable limit is one which contains more, slightly more, dissolved ammonium sulphate in this

instance, than the solution will hold as a stable solution. In other words, it is an unstable condition which will maintain slightly more than the solubility limit of ammonium sulphate for a relatively short time, and within this metastable limit there is a minimum tendency for the production of new ammonium sulphate crystals, which are sometimes referred to as seed crystals, or nuclei. And operating so that the solution as maintained within the metastable range of supersaturation tends to produce the minimum number of new crystals, and to cause the ammonium sulphate which is formed in the reaction to be deposited instead upon the pre-existing crystals which are already present in the system, the net effect of which is to produce larger crystals for a given quantity of ammonium sulphate product than if a larger number of new crystals were permitted to be formed.

Q. You used the term "a saturated solution." A. Yes, sir.

Q. Where does the variation begin between the saturated solution and the supersaturated solution? A. My understanding, your Honor, of a saturated (402) solution is a solution which at a given temperature—because the saturation limit varies with temperature—at a given fixed temperature a saturated solution is one which has a dissolved concentration equal to the concentration which will be found in such a solution at that same temperature if the solution is left for a long period of time in contact with additional quantities of the solid so that the quantity of the solid in solution is permitted to come to equilibrium with the crystals which are not in solution. In other words, any deviation from the saturation concentration will be changed to the saturated concentration if the solution is maintained in contact with additional crystals for a long enough period at that temperature.

The Court: Thank you.

By Mr. Lutz:

Q. In other words, Mr. Helm, I believe what you are saying is, then, that a saturated solution is one which would maintain a certain stability over a long period of time, and that the solution can, for a temporary period of time, contain more of the dissolved solid, and that's what we call supersaturated? A. That's correct.

Q. And the metastable range of supersaturation is (403) one rather indefinite range in this supersaturation stage? A. That is correct, and I might add that beyond the metastable range or above the metastable range there is a tendency for additional crystals to be precipitated in rather large quantities.

Q. But I understand there is no definite, clear dividing line between these different degrees of supersaturation? A. That is my understanding also; such things as agitation and other similar conditions have some effect on that limit.

Q. Now, Mr. Helm, when you were discussing the splash plate type of spray, I believe you said that Koppers has used that type of spray in a number of locations for quite a time, is that correct? A. That is correct.

Q. Approximately how far back can you date the use of that splash plate spray in coke oven equipment by Koppers? A. That type of spray was used by Koppers in coke oven gas processing equipment back as far as 1916, back to some of the earliest installations made by Koppers.

Q. I show you a copy of Becker patent No. 1,747,616, [DX 25] and call your especial attention to Figure 3. Can you (404) tell us whether Figure 3 shows any use of sprays such as you have been discussing? A. Yes, sir, it does.

Q. Where? Please tell us the location. A. One of the locations is in the collecting main for the gas leaving the ovens in the upper left-hand corner of the Figure 3, designated by the number 13—that is, the collecting main is designated.

Q. What is number 12 on that figure? A. Number 12 is the ascension pipe, or stand pipe for conveying the gas from the ovens into the collecting main.

Q. And just above the reference 13 are some lines which come down from pipe 29. What do those lines represent? A. Those lines represent a spray for contacting the gas in the oven collecting main with liquor, which is commonly referred to as flushing liquor, for the purpose of cooling the gas.

Q. And can you tell us whether or not sprays in this location are still being used by Koppers today? A. Yes, sir, they are.

Q. Have any other type of nozzles been used in this location?

(405) The Witness: Other than splash plate type?

Q. Yes. A. Yes, since sometime in the middle 1920's Koppers has used Spraco nozzles in this location.

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Q. Mr. Helm, I believe you have several times in your testimony referred to Koppers installations that were made prior to your becoming an employee of Koppers. Will you please explain how you became familiar with (427) those prior installations? A. As a part of my position as manager of the development section, as well as a part of my duties in my previous positions with Koppers Company, it has been my duty to become familiar with many of the past installations through the drawings, specifications, operating data and other records that are kept by the company for that purpose.

Q. And I believe in connection with all of these prior installations you have testified about, you have recently reviewed those drawings and other records? A. That is correct.

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Q. Now, Mr. Helm, you have testified concerning the structure of some of the old Koppers type saturators which were installed in 1917 and dates thereabout, which were of the cracker pipe type that have a horseshoe shaped cracker pipe. Can you tell us what materials were used in those saturators? A. The saturator shells were made either of steel or cast iron and were lined with lead to resist the corrosive action of the sulphuric acid in the liquor. (430) The outer shell, having little or no resistance to the acid, served only to back up and strengthen the lead lining which was the corrosion-resistant material.

Q. Now will you please tell us, give us your opinion, as to whether or not it would be very practical to use the sprays in that type of a saturator? A. In my opinion it would not be very practical as shown by the operation of the Bethlehem or Underwood spray saturators, due to the tendency of the crystals carried by the liquor, which would be sprayed into the saturator, to erode the lead. These crystals are very hard and abrasive and spraying this material against the lead lining would have a very similar action to sand-blasting material.

Q. Can you tell us what material Koppers uses today in its spray saturators? A. Yes, sir. We use type 316 extra low carbon stainless steel. I might add that we have not in all cases used extra low carbon stainless, but there is a definite maximum limitation of the carbon content of the type 316 stainless steel in order to obtain satisfactory welding and fabrication characteristics.

Q. And I believe that you testified in connection with the Seaboard saturator that Koppers first used (431) stainless steel in saturators in about that time, 1944, is that correct? A. With the exception of the cracker pipes in the Monessen and Weirton type saturators. In those cases the cracker pipes were simply made from sections of stainless steel pipe and required no welding or other

fabrication. At that time Koppers had not yet developed satisfactory welding techniques for the use of stainless steel in order that the welds would be resistant to the corrosive action of the acid-containing liquor.

Q. Do I understand this problem of welding was one reason why stainless steel had not been used earlier in the saturators? A. That is why it had not been used any more extensively earlier.

Q. Were there any other problems so far as materials or equipment go that retarded the use of sprays in saturators? A. Yes. It has only been within the past approximately fifteen years that satisfactory pumps were available of circulating liquor containing crystals in sulphuric acid. It was necessary to have available pumps made of corrosion resistant materials and provided with suitable packing glands which would prevent the (432) liquor containing crystals from scoring the shafts of the pump and from destroying the packing, thus permitting excessive leakage from the pumps.

Q. Now, Mr. Helm, you have testified about several types of saturator that were known prior to the beginning of 1946, which had a relatively low seal and were of the cracker pipe type. Is that correct? A. That is correct.

Q. And I wish you would give us your opinion of the comparison of that saturator which apparently was the best known type say at the end of 1945, compare that with the spray type saturator as to the different characteristics. First of all, how would they compare as to recovery of ammonia? A. Either type, either the cracker pipe type or the spray type would recover essentially all the ammonia. There is no difference.

Q. How about the production of crystals? A. They both produce ammonium sulphate crystals of essentially the same type and characteristics.

Q. How about the manpower required for their opera-

tion? A. For similar installation, as far as auxiliary equipment is concerned, in handling approximately the (433) same quantity of gas, or gas in the same order of magnitude, the manpower requirements would be exactly the same.

Q. How about the cost of power to operate? A. The cost of power for a low seal cracker pipe saturator as compared with a spray type saturator, would be almost the same. Calculations have been made—I have made calculations, comparing the power requirements for the cracker pipe saturator installed at Wheeling, which is the one installation which has been accused of infringing the Otto cracker pipe patent, as compared with the power requirements for the spray type saturator installed at Wheeling, which is the second accused installation, and on the basis of handling equal quantities of gas in both cases, I have estimated a difference in power requirement of between two and three dollars per day. This includes the power required for pumping the gas through the seal in the case of the cracker pipe saturator, and includes the cost of pumping the liquor in the case of the spray saturator.

Q. Would you say that difference in power cost was substantial? A. I would say it is insignificant.

Q. How about the original installation cost of (434) these two types? A. We have compared actual installation costs of spray type saturators and cracker pipe saturators of similar capacities, correcting the costs to bring them in line as far as changes in equipment costs from the time one installation was made to the time the other installations were made, and there was very little difference in cost for installation to handle the same quantity of gas.

Q. In summary, what would you say are the chief advantages of the spray saturator? A. I believe the chief

advantages of the spray saturator as compared to the modern cracker pipe saturator are ease of operation in the case of the spray saturator, and to some extent less ground space requirement for a given gas capacity.

Q. Well now, will you please explain just a bit more what you mean by ease of operation? A. There is a substantially less tendency for the gas space of a properly designed spray type saturator to fill up with depositions of ammonium sulphate or rock salt than is the case with cracker pipe saturators, so that it requires less attention from the operating personnel to maintain the spray type saturators operating satis (435) facterily, in my opinion.

Q. I infer from your last answer, Mr. Helm, that there is some tendency of even the spray saturator to salt-up. A. That is correct. Primarily in the spray nozzles and the connections thereto.

Q. And I believe it is the practice in operating Wheeling, and other Koppers spray saturators, to use a periodical kill to remove this tendency to clog the spray nozzles. A. That is correct. It has been found that by killing the bath, which means adding sufficient water and acid to completely dissolve all the crystals in the system, that the depositions of crystals in the sprays can be dissolved before they become serious.

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Cross Examination by Mr. Morton:

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(462) Q. I show you United States patent 2,671,011. Is the patent I have just handed you a patent owned by the Koppers Company? A. Yes, sir, it is.

Q. And is it a patent which discloses a construction which is embodied in the Follansbee installation? A. Yes, sir, it does.

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Edward J. Helm—For Defendants—Cross.

Q. Thank you. Now in your capacity as manager of the development department, you are familiar, are you not, with the construction of all the saturators that have been installed by the Koppers Company at least since the installation of the center dip saturator at Wheeling? A. I believe that is correct. I would not say that I know all the details but I have some familiarity with all of them.

Q. I believe I am correct that the first saturator called the spray saturator for general purposes, that the (479) Koppers Company installed was at Neville Island in the premises of Pittsburgh Coke and Chemical? A. That is correct.

Q. And that since that time all of the ammonia saturator apparatus which has been supplied by Koppers to its customers have been spray type. A. I believe that is correct.

Q. Tell us whether all that apparatus has been of the type exemplified by the Wheeling installation, model 17? A. No, sir, there have been some installations which have differed in some respects from the Wheeling installation.

Q. And one of the differences, for example, has been that in some of these more recent; that is, more recent than Wheeling and Neville Island installations, some of the apparatus which is physically combined with the geometric structure arrangement shown in 17 have been separated again. Isn't that correct? A. That is correct. One of the elements incorporated in the Wheeling saturator on one or two installations, has been supplied as a separate element.

Q. For example, some of your recent installations have the acid catcher as a separate vessel, similar in (480) that respect to being a separate vessel to the arrangement

of the acid catcher on exhibit 15. A. That is correct. Different in form but similar as being a separate unit.

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Q. Now let us turn a moment to the installation (496) about which you talked for some time yesterday, namely, the Underwood modification of the old style cracker pipe saturators at Bethlehem plant of the Bethlehem Steel Company. It was my understanding of the purport of your testimony yesterday that you identified the exhibit drawing, specifically Defendants' Exhibit 11, as illustrating a construction which was installed at Bethlehem. Did you ever see that construction yourself?

The Court: Isn't that number 14? You are speaking of the drawing rather than the exhibit.

Mr. Morton: Yes. The actual drawing, I believe was exhibit 11.

A. I presume that is the right number. No, I did not see this modification in operation at Bethlehem.

Q. So that the exhibit, the simplified exhibit 14, which was I believe prepared under your direction, was prepared as a simplification of the drawing exhibit 11, as a matter of engineering experience, and not personal experience with the apparatus? A. That is correct.

Q. It was, I am sure quite accidentally, in the phrasing of the questions and answers, the implication that you knew that this drawing was a correct drawing of an installation that was made, and any knowledge you (497) have about that is something you have learned in your investigation of this litigation. Isn't that right? A. That is correct. I might add, Mr. Morton, that I had learned of this drawing prior to the instigation of this litigation.

Q. You had? A. Yes. Not through observation, but I had learned of the existence of this drawing and had seen this drawing.

Q. Prior to the instigation of the litigation; might I ask, had you seen that prior to June 3, 1952, the date of the issue of the patent in suit? A. No, sir.

Q. Where did you first see the drawing exhibit 11? A. I first saw the drawing exhibit 11 at Bethlehem, Pa.

Q. So far as you know, were you the first employee of the Koppers Company to see that drawing? A. I do not believe that I was.

Q. Do you know who was? A. I do not know who was first.

Q. When did you first hear about the existence of the apparatus depicted or purported to be depicted, on exhibit 11? (498) A. I do not know whether I can answer with any certainty as to the exact date. I believe that I heard of the existence of such an installation, without knowing all the details of the installation, prior to the issuance of the Otto spray patent, but I am not in a position to testify to that with certainty.

Q. Do you know whether the existence of this installation was known to you prior to the time when the Neville Island saturator built by Koppers was designed? A. Obviously, if I can not testify to knowledge of it prior to the issuance of the Otto patent, I can not testify to knowledge of it at an earlier date.

Q. You were familiar with the project of designing the Neville Island installation, were you not? A. I was not directly connected with the design at that time, but I have made a study of the events that occurred at that time on the basis of records available in the Koppers Company and the Neville Island plant of the Pittsburgh Coke and Chemical Company, and through discussions with Koppers engineers who did participate in that design.

Q. Did your study lead you to conclude that any use was made of the disclosures on the drawing exhibit 11 (499) in the making of that design? A. The broad principles of

spraying saturated ammonium sulphate solution containing crystals and free acid into the unobstructed gas space was utilized as incorporated in the Underwood saturator. It was incorporated in the design of the Neville Island saturator. The details of carrying out those principals were substantially different.

Q. That is not exactly what I was inquiring. What I want to know is, did your investigation of the circumstances of the design of the Neville Island installation give you any reason to believe that the designers of that installation had knowledge of Mr. Underwood's experience? A. Yes.

Q. What were the indications that led you to that belief? A. The man who was primarily responsible for the conception and the design of the Neville Island saturator, Mr. VanAckeren, told me that he had been familiar with the Underwood spray saturator operation.

Q. Did he tell you when he acquired this familiarity? A. He told me that he saw the installation in (500) operation during its period of operation.

Q. Mr. Joseph VanAckeren is the VanAckeren who is the patentee of patent 2,671,011 which has been marked Plaintiff's Exhibit 27, is he not? A. That is correct.

Q. And he is also an officer of the Koppers Company? A. Pardon me?

Q. Am I correctly informed that he is an officer of the Koppers Company? A. Not at the present. He is retired and retained as a consultant.

Q. In the past he has been an officer of the Koppers Company, has he not? A. He has been a divisional vice president. I am not certain of the legal terminology, whether that constitutes an officer of the Koppers Company or not.

Q. The division of which he was vice president was which division? A. The engineering and construction division.

Q. The one in which you are employed? A. That is correct.

Q. In your examination of the saturator installations which were made by the Koppers Company, and the designs, I take it, and the like that were on the drawing boards (501) of the Koppers Company, did you happen to run across any indication that the Koppers Company submitted a bid or proposal to the Bethlehem Steel Company for the erection of a saturator to replace the Underwood modified saturators? A. I do not believe that I can recall seeing any documents which would indicate that, but it is my belief that they did enter such a bid.

Q. That replacement, as I recall the testimony of Doctor Otto, was completed in 1946, and I believe that the normal length of time would indicate that the contract negotiations would have been undertaken some time in the preceding year. Am I right? A. I would think that is right.

Q. Now can you tell me in 1945, or about that time, what sort of saturator installation Koppers was offering, specifically what sort it offered to Bethlehem? A. I can not tell you specifically what type was offered to Bethlehem.

Q. At about that time Koppers offered to install at Wheeling a center dip type saturator, did it not? A. I believe that was a year or two, or three later.

Q. Later. You don't have any doubt that Koppers (502) did not offer to replace the Underwood converted or modified cracker pipe with spray saturator? A. I have no doubt that they did not offer.

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Q. I believe you testified yesterday that one of these bustle-type saturators was operated for a brief period of three weeks, I think you said, after its initial installation with the low seal. Was it this one? A. I have forgotten now. Is that the—

Q. That is Seaboard? A. No, it was the Aliquippa saturator.

Q. The Aliquippa saturator is the one illustrated on Defendants' Exhibit 30. If I recollect correctly, you said that when this was operated with zero seal ammonia recovery was on the order of 97½ per cent? A. That is my recollection also.

(515) Q. It is a fact, is it not, that in standard operating practice, such as, for example, the operating practice of the Wheeling Steel Corporation here at Follansbee, both with its cracker pipe saturators and with its spray type saturators, that we can obtain, as a matter of course, ammonia recovery better than 99 per cent? A. That is correct.

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Q. Do you know whether Koppers Company has a license under the Jeremiassen patent? A. No, sir, so far as I know, Koppers does not.

Q. Now, you were asked yesterday how you would describe the solution passing from pump 1 around this large pipe and coming down into the funnel of Jeremiassen, and I believe you said in essence that you would describe that solution as the same sort of solution, the same solution in fact, that was found passing in the circulation of the Wheeling saturator. Do you recall you so testified? A. Yes, sir.

Q. Now I will ask you a little different question. I am going to ask you how did Jeremiassen define that solution? * * * A. So far as I have been able to determine from a quick study, I believe that he describes the solution as being maintained within the metastable limits of supersaturation at all points in the system.

Q. He says, does he not, that the contact of this solution coming through here with the crystal suspension 7 serves to release all of the supersaturation, so that as it flows from here, back around and down, at no time does the degree of supersaturation exceed the metastable limit. Doesn't it necessarily follow that if this metastable limit condition is

one which is released by the presence of crystals, that it can not exist in the (534) presence of crystals? A. I believe, Mr. Morton, that is a matter of degree. The major part of the supersaturation is released in passing upward through the crystals in chamber 11. Is that correct? Yes, in chamber 11. But it is not necessarily true that the solution flowing upward through the pipe beyond pump 1 would be completely freed of supersaturation. However, whether that is so or not has no bearing on whether crystals are carried mechanically by the solution beyond the pump.

Q. Well, if I understand the terms as Mr. Jeremiassen describes them, the state in which the liquor is which he calls metastable is defined as that state to which the supersaturated liquid comes after it has been subjected to the releasing action of a body of crystals, so quite evidently, it seems to me that it can not carry its own crystals with it, so it would be released as fast as it got there. A. That is not at all my understanding of his definition, and I do not believe you have the right concept, Mr. Morton.

Q. I asked you to point out to me anywhere in Jeremiassen's description any statement that any portion of the circulating liquid contains crystals except those (535) portions which he shows to be speckled in both figures. A. So far as I know, there are no statements in the specification which so state, but I believe that it is a matter of engineering knowledge that within the practical limitations of design of equipment of this type, it is impossible to pump a large circulation of liquor as specified in the Jeremiassen patent through a crystal container such as chamber 11 without entraining crystals into the outlet line, unless chamber 11 is made impracticably large.

Q. That is what you think would happen if this were operated? A. That is correct.

Q. You have no knowledge of whether this particular apparatus ever was operated? A. Not in the exact form shown on the Defendants' Exhibit 24.

Q. Was it ever operated in the form shown on the figure of the patent itself? A. I don't know, but I do have knowledge of equipment operated by Koppers and by others, embodying all the essential features so far as process relationships are concerned, in which crystals always have been entrained in the liquid leaving the top of the (536) crystallizing chamber.

Q. Are these apparatuses operated by Koppers, and others of which you have knowledge, operated always to maintain the circulated liquor within the metastable limit? A. No, they are not.

Q. Then they are not operated the way Jeremiassen describes his operation, are they? A. That is correct, but in my opinion that has no bearing upon the physical impossibility of pumping large quantities of liquor through crystals without mechanically entraining the crystals.

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(587) Q. Just one more question, Mr. Helm. In your familiarity with the series of saturators that have been built by the Koppers Company over the years, are you able to tell me what were the last types of cracker pipe saturators, or the last type cracker pipe saturator, offered by the Koppers Company to its customers? A. So far as I can recall, the last type was the Wheeling bell-mouth type.

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Re-direct Examination by Mr. Lutz:

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(592) Q. And I believe you have previously identified the spray nozzles which are in the primary absorption chambers. Please point those out again at this time. A. Those are the 14 nozzles shown at the top of each of the

two primary absorption sections and they are shown extending downward at an angle from the top of those two primary absorption chambers.

Q. Now, Mr. Helm, referring to these 14 spray nozzles which you say are indicated in the primary absorption chambers, will you please tell us whether or not liquor normally circulated through those spray nozzles in this Wheeling spray saturator contains crystals? A. The liquor normally circulated through the spray nozzles in the primary absorption chambers always contains (593) crystals, except during the period between the kill and the formation of the first crystals after the kill period.

Q. How do you explain the presence of these crystals? A. Those crystals are present because it is practicably impossible to remove them from the circulated liquor and for that reason, it is impossible in apparatus of a practicable size to prevent having them in the circulation.

Q. Will you please tell us whether or not there is any particular advantage in having crystals present in the liquor circulated to the sprays? A. In my opinion there is no advantage in having crystals present. It is merely a case of being impossible practicably to avoid having them present. There might be, from a theoretical point of view, a slight disadvantage in having crystals present, but certainly no advantage, in my opinion.

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ARTHUR H. RADASCH, having been first duly sworn as a witness, thereupon testified as follows:

Direct Examination by Mr. Morton:

Q. Will you tell the Court what position you now hold? A. I am Professor of Chemical Engineering at Cooper

Union in New York, and head of the Department of Chemical Engineering.

(625) Q. What is Cooper Union? A. Cooper Union is a privately endowed educational institution.

Q. What degrees are awarded by your department? A. Well, degrees are not awarded by the department, but students who are graduated from the course, supervised by our department, get the degree of Bachelor of Chemical Engineering.

Q. And Cooper Union is located in New York City? A. Yes.

Q. And its authority to grant degrees, of course, stems from the State of New York, does it not? A. Correct.

Q. What degrees do you yourself hold? A. I have a degree of Bachelor of Science.

Q. And are you a registered engineer? A. Yes, sir.

Q. In what state? A. New York.

Q. To what professional societies do you belong? A. The American Institute of Chemical Engineers, the American Chemical Society, American Society for Engineering Education.

Q. Prior to embarking upon an academic career as professor of chemical engineering, did you hold any (626) position in the chemical engineering industry? A. I was a chemical ergineer with the Barrett Division of Allied Chemical & Dye from February, 1927, to October, 1938.

Q. In the course of that practical experience did you have any experience with the problems of ammonia recovery? A. I did a little in 1930 and '31.

Q. And did that experience require you to make any reports on the subject to your employer? A. Yes, sir.

Q. I don't believe you told us when you got your own degree. A. In 1920.

Q. From what institution? A. Massachusetts Institute of Technology.

Q. You have been retained by the plaintiff here, Dr. Carl Otto, to serve as an expert witness in this case, have you not? A. Yes, sir.

Q. And in preparation for that task you were supplied with a considerable body of prior art and other material relating to the subject matter of this case, were you not? A. Yes, sir.

(627) Q. And did you supplement your material with which you were supplied by Dr. Otto and his counsel with some investigations of your own? A. Well, yes, in addition to the material supplied me I read a number of literature references, and I guess checked up on some other patents; mostly with, however, technical literature.

Q. Now, Professor, did you embody your conclusions as to the line of development which had been generally followed in the evolution of processes and apparatus for the recovery of ammonium sulphate from coke oven gas onto a chart of some sort? A. Yes.

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Q. Now, would you explain to the Court, Professor Radasch, what you intended to illustrate by this chart, [PX 32], taken as a whole? A. Well, your Honor, I read a lot of these patents, and I felt it was necessary to get some rhyme or reason, and I tried to develop an analysis here which would bring them all together, show similarities and differences. In making (628) that analysis, they are all dealing with a general problem which is illustrated in the diagram on the left-hand side there, which is coke oven gas containing ammonia flowing in and being treated in some kind of a process with sulphuric acid to make coke oven gas not containing ammonia and to make ammonium sulphate.

The Court: When you speak of similarities and differences, do you mean similarities and differences as between these various drawings or—

The Witness: The various schemes and the development of ammonium sulphate production over the years, as represented by—

The Court: Similarities and differences in the process, is that what you mean?

The Witness: That's correct, sir.

Q. (By Mr. Morton:) Now, to recap, Professor, I think the general process which you have illustrated by this figure on the left is common to all of the varying apparatuses for the production of ammonium sulphate from coke oven gas, is it not? A. Yes. They all take coke oven gas with ammonia, get the ammonia out by using sulphuric acid, and they make ammonium sulphate.

Q. Very good. Now, from there. (629) A. In making this analysis to analyze what goes on inside the circle, so to speak, since they deal with gases and since they deal with liquid solutions, and since they also deal with solids or crystals, it seemed to me that I should analyze this on a scientific basis and consider those factors which are necessary to characterize systems of that kind, and the three factors which the physical chemists use, and the chemical engineers also, are the pressure, temperature, and the various concentrations that are involved in these systems.

I had to select one of these factors as the principal distinguishing characteristic to get an illustration such as I wanted. Pressure is not a particularly good one, because except for certain parts of cycles they all operate at substantially atmospheric pressure. There are little differences in temperature, although they all on these are operating say from 45 degrees up to 80 to 85 degrees.

Q. And by that you mean 45 to 85 centigrade? A. Yes. The best division was in terms of the concentration, so I have drawn a line right across the middle of this chart

from right to left, and I have above that line a number of processes which we have been talking about, or which have been talked about here, which use (630), as stated in the upper left-hand corner, saturated and supersaturated solutions of ammonium sulphate containing acid, which are introduced into contact with the gas in some fashion to accomplish the general objective, the common objective.

Below that line are others which have been mentioned, a couple of them, and a couple of others that have not been mentioned, which use unsaturated solutions to accomplish this common objective of making ammonium sulphate.

I could probably next, if that is clear, start talking about these things somewhat chronologically, if that is agreeable.

Q. Before we proceed with that, the color code used here is such that, as I understand it, the red, which appears on all of the four top panels but on only one of the lower row of panels, illustrates places where there is found a concentrated solution of ammonium sulphate, a saturated solution of ammonium sulphate, containing crystals? A. That's right.

Q. And the yellow which appears on all of the lower line and at two places in the upper line of panels, illustrates a solution of ammonium sulphate containing sulphuric acid but not containing crystals? (631) A. That's right.

Q. And the green illustrates gas? A. That's right.

Q. Very good. Now, would you continue with your analysis of the results of your investigation. A. Well, since this Otto patent involved spray means for doing this job, our search disclosed a couple of early German patents, which are in the lower left-hand panel. The Neumeyer German patent 24,511 in 1883 is substantially as shown here, that is, quite close to the diagram. There was a vertical pipe with holes in it, which is somewhat like the pipe

that was used by Underwood at Bethlehem, and acid containing some ammonium sulphate was put in that pipe and squirted out of that pipe, hitting the sidewalls, ricocheted, and formed more or less of a spray.

There was another patent by Wellstein the following year, also a German patent, which was somewhat similar, except that he had a pipe at the end of which he had a lot of little holes and squirted a spray or a mist out of that grouping of holes, but essentially it was the same as the other one.

There was another patent, noted on the bottom of the panel, by Brunck, which is a United States patent, in (632) 1906, again which was very similar.

All of these had only solutions, two solutions in the tower, and they made their product of ammonium sulphate salt somewheres else.

Q. That is to say that the solution that they sprayed in or otherwise dispersed into the gas in the tower was not a crystal-containing solution— A. That is correct.

Q. (Continuing)—and was, in fact, not even a saturated solution? A. That's correct.

Q. So that the production of crystals was reserved for a subsequent operation not taking place in the apparatus shown, is that right? A. That's correct.

Q. All right. A. Then in the panel just above it is this early Brunck patent 1904,—that's the year 1904—which was an early cracker pipe design. It's a little hard to show this thing in just the one view such as I have here, but it consists of a gas inlet pipe, shown on the right, and a gas outlet pipe, shown on the left. Then in order to get from the inlet pipe to the outlet pipe the gas had to pass down under a baffle which has these notched edges, (633) and there were a series of these baffles along, one behind the other, so that within a compartment on one side it passed

under the notches and got into the other side, and then that was in a compartment connected to the outlet pipe.

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(636) The Otto patent 2,599,067 is, of course, the patent in suit and is the subject matter of Plaintiff's Exhibit 1. The Doherty patent 997,908 is a part of Defendants' Exhibit 18, and also of Defendants' Chart 19. The Becker patent 1,307,534 is part of the Defendants' Exhibit 18 and also of Defendants' Chart 21. The Tiddy patent 2,368,901 is contained as a part of Plaintiff's Exhibit 2, and was one of the references considered by the Patent Office.

Q. Now, Professor, I interrupted you when you were explaining the operation of this Brunck invention in patent 773,784, which we have now given the number Plaintiff's Exhibit 31. Would you continue with your explanation of your analysis. A. Well, this structure, which is shown only in diagrammatic form of the inlet and outlet pipe and these cross members with the serrated edges, the outside of that structure extends down a little farther into the bath so that this whole thing is open underneath, and this is, you might say, the granddaddy of all of these cracker pipes, and I believe in those days that they got the ammonium sulphate out by just putting a hole in there into this open bath, or rake or something, and just raking them out, but it did use a concentrated acid. It operated (637) on hot raw gas, that is, above—about 80 or 85 degrees, and it used these open baths which were desired to avoid these stoppages.

The next thing historically is the Doherty one, 997,908, which is down below. There was a better diagram of that that the Koppers people made that might be helpful to look at at the same time.

(638) This large exhibit over here, 19 I think it is, is a reproduction of the drawing in the patent. The only thing I would comment about it at this time is that the sprays

sprayed downwards into the gas, also flowing downwards. In other words, it was a concurrent flow, and then there are these bubble caps which act in a reverse direction at the bottom, something like a baby cracker pipe acting in the reverse direction, or in the same direction as these cracker pipes which are in some of the saturators where the gas comes from the outside and goes inwards, underneath rather than coming from inside and going outward.

Q. In the Doherty device are there any crystals present in the area where the reaction between the ammonia gas and the acid which causes it to take place? A. I think not because he took the solution out of that tower and took it somewhere else to evaporate and produce crystals.

Q. Would a bubble cap arrangement such as shown in the bottom of the Doherty arrangement on your chart Plaintiff's Exhibit 32, or on the enlarged drawing which is Defendants' Exhibit 19, would that sort of arrangement in your experience as a chemical engineer be a suitable apparatus if it were intended that crystals be (639) formed at that place? A. No. Ammonium sulphate crystals are pretty heavy and they just settle to the bottom of the solution and this plate on which these bubble caps rest, or over which they rest, would simply fill up very soon with salt and that would be the end of the thing.

Q. In other words, gas would not run through it for any appreciable period of time without clogging. Is that right? A. Yes, that's right. Now the next one I might mention, I guess, is the one to the right of it, which is this Becker 1,307,534. This was distinguished from some others by being a hot wash tower operating at 80 degrees. I believe this was the patent where there was some discussion as to whether there was any precipitation of ammonium sul[illegible] in the tower. I would like to get my copy. Referring to the patent, I would like to read those two sentences again, the first one which says, "The gas both enters and leaves

the acid washer with a temperature that is still somewhat above its water saturation point, but so little above that practically no water is taken up by the gas from the acid solution, and consequently there is no (640) precipitation of ammonium sulphate in the acid washer tower."

There is a little bit more, but the next sentence says, "The acid solution from the said acid washer tower 8 flows into a saturator 9 carrying with it into the saturator any slight amount of ammonium sulphate that may have been precipitated."

I think Mr. Helm interpreted that to mean that there could be—that this tower could operate so that crystals would be present, and I think in the examination by Mr. Morton he suggested that possibly the tower might get out of control and cause that. Well, I agree to that, it is a possible explanation but I would like to offer another one as to why this second sentence was inserted in the Becker patent.

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(644) Q. Let me ask you this. Is Mr. Becker's putting in the second sentence consistent with your understanding (645) as an engineer of how a piece of scrubber apparatus operates? A. Yes, sir. I think these diagrams which are in patents, and which have been reproduced on these charts, are essentially diagrams somewhat idealized, and the one I have presented is even more idealized. Now a tower is not an idealized piece of apparatus. It is a real piece of apparatus, and there is no use in thinking the solution which you admit to the top of the tower descends in some kind of a mathematical plane and starts off at some concentration and every foot picks up a certain definite percentage all the way across the tower and exits in any condition such as a concentration is the same in a mathematical plane all across that tower. A tower is a real thing, and this is the kind of packing that there is in a

tower. I don't know whether this would be used in a sulphuric acid tower but it is the only piece that I could get hold of. If this packing is in there and the liquid descends, it can get hung up, and there can be local situations as Jeremiassen described, where even though you plan to operate an exit with an unsaturated solution, this hanging up of the liquid in there in contact with more gas than corresponds to the liquid flow, that liquid (646) can become saturated and finally supersaturated, so that salts can be produced in a tower, and I would like to explain the second sentence, that that would account for Mr. Becker's thinking that even though he wanted to run a tower so that he came out with an unsaturated solution, this salt which might be temporarily formed could become dislodged, could dump, could be partially dissolved so that the encrustations could no longer hang on to this thing, and could drop down into the exit solution.

Now that is a rather long explanation but I thought I could shorten it. So that is my reason for putting Becker 1,307,534 below the line.

Q. That is below the line which separates apparatus working with a saturated solution containing crystals from those working with unsaturated, non-crystal containing solution? A. Yes, sir. In this particular patent, and also in the preceding patent issued on the same day, the product of the towers went to another place for production of the ammonium sulphate crystals. While I have made this whole analysis on the basis of a first approach in terms of fundamental science, I do recognize, of course, that there are, comparing one with another, a great many (647) differences in the way a man would carry out his idea, and so therefore there are many engineering differences here besides differences in concentration.

Now I can go to the Becker patent in the second panel at the top.

Q. Does that tentatively illustrate anything more than a conventional cracker pipe? A. Well this particular sketch here is the type of thing shown diagrammatically in the earlier 1919 Becker patent, but in my thinking this particular conventional cracker pipe stands not only for 1919, it stands for 1930, it stands also for multiple cracker pipes, and all kinds of cracker pipes. I do recognize, of course, that there are engineering and operating differences one from the other. And it stands also for the oval cracker pipe saturator.

Q. Now in the cracker pipe design which you have idealized under the heading Becker 1,747,616, does gas pass through the reaction zone from inlet to outlet unobstructed in its flow? A. No, it is obstructed.

Q. Obstructed by what? A. By the liquor that it would have to pass through and push out of the way to do so.

(648) Q. That is the gas is introduced into a bath of liquid and bubbles through the liquid before it can proceed to the exit? A. That's correct.

Q. What is that bath? A. Well, that bath, since this represents the conventional cracker pipe design, when it is operated normally, will be a solution of ammonium sulphate that will contain also sulphuric acid and in which there will be suspended ammonium sulphate crystals.

Q. That is why it is placed above the line? A. Yes, sir. Now I come next to the Jeremiassen patent, and I think an understanding of the Jeremiassen patent depends absolutely on a clear understanding of what is meant by these metastable solutions.

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(649) The Witness: I hope I can do this without adding to the confusion. I would like to simplify it if I can. Here are a couple of solutions I prepared in New York, which are in the metastable con-

dition. Now I think we ought to have a definition of that. As I understand it, a metastable equilibrium is a very definite equilibrium. It is not the most stable one but it is a very definite one, and can continue to exist for usually as long as you want it, and it can be discharged when you introduce a stable phase. There are several materials that can be used to demonstrate this, and one of the best is sodium thiosulphate, which is the material developed in water in each of these two flasks.

Let me open one of them. And then I got a bottle of sodium thiosulphate crystals here and if I take a little tiny seed, all I have to do is crack up one of these and get a little material. I rather hesitate to do this, because sometimes these experiments don't work, but let's see if I can make this work. If not, I will have to try another. Drop a little seed in there. You can see that grow and of course now the metastability is destroyed, and that is what happens when you introduce a stable phase.

Mr. Lutz: Let the record show that Professor (650) Radasch not only dropped a crystal, but shook the bottle.

The Witness: May I use this other bottle, and ask Mr. Lutz if he will hold it and not shake it or do anything he wants to, and I will put another crystal in it.

Q. Before we come to that, you might tell the court just what is in the bottle before you that is perfectly clear. A. That is a metastable solution of sodium thiosulphate in water.

Q. In other words, that is a solution of sodium thiosulphate which contains more sodium thiosulphate in percentage terms than is considered to be a saturated solu

tion. Is that right? A. If you took the solution off this and filtered off the crystals that would be a saturated solution and the content of sodium thiosulphate in that solution is less. These are both prepared from the same original mixture, because some of it will have been removed as crystals.

Q. The witness has reference to the bottle in which he dropped a seed crystal and which now contains a very substantial mass of crystal. Those two solutions, the (651) one you have just treated, and the one in the sealed bottle, were made by you in New York? A. Yes.

Q. And were they brought down by you in what sort of transportation? A. Shall I name them all?

Q. Yes. A. Foot, subway, bus, train and private automobile.

Q. You came by way of Steubenville and drove over here. Did you take any extraordinary means to protect these two little bottles from agitation while you made this trip? A. No, you don't have to do that. I think there is a lot of misunderstanding about this metastable state and I am sure that I can read from the Isaachsen patent we have already read from, on page 2, line 35—I am sure, of course, Issachsen never used the word metastable, but he is talking about this condition. He says this supersaturated condition is completely stable; that is this one where it is completely stable in the sense that the supersaturation is not released even by very small whirls and the presence of other solid bodies.

Q. By other solid bodies, he means what? A. Well, I have put sand in a solution of this kind (652) and it does not discharge it.

Q. You mean the solid bodies other than particles of a dissolved solid. Is that right? A. That is correct. Now I would like to read one more sentence, and I would like to insert two words, and just so the record is clear, I will

use parentheses where I insert the words. He says, "This distinguishing feature that it is possible to prepare the solution at one point (New York) and then to carry it far away to another place (Wheeling) where the supersaturation is released has not hitherto been so clearly recognized, observed and measured as to lead to technical conclusions, although it is of the greatest importance for the industrial production of large crystals."

In other words, the fact that you can do this is not clearly understood by a lot of people. Now, to me, when Jeremiassen uses the word metastable, and he uses it in six places—I have marked them. One is page 1, column 2, first line; the second one is line 20; the third one is line 45; the fourth one is page 2, column 1, line 43, and the fifth one, line 60, and the sixth one, column 2, on that page, line 2. He uses it six times and in the third case, he defines it, and that is the one on page 1, line 44, or thereabouts. "This results (653) in the liquid becoming supersaturated to within the metastable limit, namely, to a degree of supersaturation at which no crystals are formed."

Q. You indicate that it is in a condition similar to that supersaturated metastable solution in the clear bottle. Is that right? A. This one here, the clear solution, is a metastable solution but the content is within the metastable limit, which is somewhat above the solubility here in the saturated solution, and in the other three places, four, five and six, while he doesn't re-define it, because you only have to define it once, he defines it by inference, by telling what will happen if you don't have this; for example, in place four, he says, "At no point will it rise above the metastable limit so that the holes in the bottom 3 will not be obstructed" by this sort of thing.

Q. You mean by the crystal formation that took place in the little bottle when you added to that clear solution a seed crystal. Is that right? A. Yes, and in place five,

he says, "Which are at all points within the metastable limit so that coatings, crusts, on the parts of the apparatus are avoided", and in place six, he talks about results in the formation of (654) fine salt and coatings caused by the limit of metastable supersaturation being locally exceeded, as explained above.

This local exceeding is the thing that I tried to explain in terms of what would happen as a solution flowed down a tower.

So regardless of what anybody as an engineer may think as to the possibility of getting this sort of thing, and I might say that it would be quite difficult myself as an engineer to do that; nevertheless that is what Jeremiassen wanted, and that's the way he wanted to operate, always this way. Now this will last a long time. I don't know how long, but from Monday to today is three days, and it will last three more days, and it will last ten years if you leave it alone and you can shake it, and that's the kind of thing that Jeremiassen wanted to operate with, and in terms of a definition, a metastable solution to me has to be this sort of thing. That is one which contains more product dissolved in it then corresponds to the saturation value that you would find if you went to a handbook. Metastable means, of course, what a lot of people say it means; that is, a percentage of supersaturation which is just a number. It is a quantity factor but metastable also means this thing (655) here which is a clear solution, which is a quality factor, and Jeremiassen, or anybody else, in talking about supersaturated solutions as they were—this of course is not always true when you say supersaturation—but when you say supersaturation within the metastable limit you mean this kind of a solution. I am talking about a quality as well as a quantity.

Q. Can the quality which Jeremiassen defines as metastable exist for any appreciable time in the presence of un-

dissolved solid material of the sort which is dissolved? A. No. When you talk like that, that is just an incongruity. You can not talk in the same breath about a metastable solution having any crystals present. It is an incongruity, an inconsistency. This is a metastable solution and as soon as you have any crystals put in it, this is what happens.

Q. When you say "this" in the first place, you refer to the sealed bottle which remains clear, which you say you prepared in New York, and now you refer to another bottle, which contained, as I understand it, an identical solution. Is that right? A. That is correct.

Q. And you added to it a little seed of the (656) dissolving material that is sodium thiosulphate and it now contains crystals and also liquid. Is the liquid in that bottle in a metastable state? A. No, sir. I would say by this time—well, the minute I put a crystal in there, it was no longer in a metastable state.

Q. And what would the proper term be to define the state of the liquid in the bottle which now contains crystals in considerable quantity? A. Well, now, about now, or soon after now, the liquid there is a saturated solution.

Q. The liquid will be saturated and what is out is simply solid crystals. It contains two things: a solution which is saturated, and additional undissolved crystals. Is that right? A. Yes. Now when I say saturated, of course, I have in mind the fact that we get it at some exact point of saturation and it would take an infinite time, but this could be analyzed almost any time now and again tomorrow, and the result would be very close. Whatever this concentration is, that is very close to saturation in the solution part.

Q. Having come to that value from above, it would be slightly above saturation at the moment? (657) A. It would be slightly above.

Q. Had this been reaching saturation by adding more and more, it would be slightly below. A. That's correct.

Q. Until you hit a point where no more would dissolve. A. That's correct.

Q. But for practical purposes, no matter which way you approach it, that liquid is not a saturated solution of sodium thiosulphate. A. That's correct.

Q. Turning then to your chart, Plaintiff's Exhibit 32, which has been marked, to illustrate your explanation of the development of these patented processes, on that colored diagram, the yellow material, the yellow areas, contain solutions of ammonium sulphate containing sulphuric acid, which is described by Jeremiassen as being in the metastable condition, and that means that it is not crystal containing. Is that right? A. That is correct. My understanding of what he wanted by way of his process, and that was the essence of both his and Isaechsen's inventions.

Q. And the red area at the bottom is where he keeps his body of crystals, the effect of which is to skim off (658) the supersaturation each time the liquid goes by. Is that right? A. Yes, in the central downcomer he wants this sort of thing.

Q. You mean the crystal clear solution? A. Right, and he wants to discharge it where you have shown the red this way, outside of that pipe.

Q. So he doesn't build up a body of crystals? A. Yes.

Q. Does he state that he operates throughout his circulating system except where he discharges for supersaturation in the red area within the metastable range in his patent? A. He says that because when he returns this liquor by the pump to the top he does not want any plugging of those holes there. He does not want any crystals like this present. He wants to operate this way.

Q. If he had crystals present, he would not be operating in the metastable range, as you understand that term? A. That is an incongruity in my thinking.

Q. Now will you explain to us the panel in the lower right, which is headed Tiddy, 2,368,901? A. Well, Mr. Tiddy worked on this for quite a long (659) time, and this was a patent that he filed in 1940, issued in 1945. His analysis of how to go about improving the saturator was a good one from the standpoint of an engineering approach, which is what some of these other people have done also, and that is to realize that there are two things going on in an ammonia saturator.

Q. What are those two things? A. Those two things are the absorption of ammonia, and its subsequent reaction with sulphuric acid to form the ammonium sulphate in solution, and the second thing is the production of crystals of ammonium sulphate. Now ordinarily a chemical engineer would not do those two things together. They should be separated, but they have been unified for a great many years, I guess starting back with the first cracker pipe, but in Mr. Tiddy's attempts to improve the operation of the saturator he analyzes the problem, and he said they should be separated, so he devised this process represented by the lower right-hand corner. He did not use the same language as Mr. Becker in 1,307. He did not say no precipitation of ammonium sulphate in tower. He said avoid saturation of solution in tower. But it amounts to the same thing. I think he probably also realized that it would be difficult to operate the tower without (660) getting locally some over saturation, and I would call your attention particularly to the percentages by weight of solution shown at the outlet 42 to 47 percent by weight of ammonium sulphate, and then over in the lower right-hand corner, 45.7. He adds a little more sulphuric acid and he gets it to 44.7 going into the top. All of those numbers are very close to each other, and therefore the thing is very close to the saturation point, and you would expect that he would have occasion crystals formed in the tower, and I understand that is actually what happens.

Q. By the way, what does this X in the middle of the tower represent? A. That is the conventional means of indicating the height of packing in a packed tower.

Q. When this Tiddy operation slipped over the edge of desirable working with undersaturated solution into saturated solution, would that be productive of operating difficulties? A. Yes. His tower would get locally plugged up. He might even get some sulphate getting in the line and interfering with movement of the fluid in the line, and he would probably have to do what he should not have to do if he operated with an unsaturated solution, that is, (661) he would have to flush out the tower.

Q. There is no question that this apparatus as illustrated in this panel is intended to operate and has a two step operation, and operates in the reaction zone with undersaturated liquor? A. That is correct, and he makes his crystals in another subsequent step, and in this particular case his was a little different from what others used. He used a vacuum evaporator.

Q. Now we come to the Otto patent in suit, as illustrated in the upper right-hand corner of the panel. I note you class that with the processes above the line. A. Well, I classify it with processes above the line because in this apparatus sprays are used which spray a saturated solution. I know that the patent says unsaturated solution can be used, saturated solution and supersaturated, but essentially it is the spraying of a saturated solution with entrained crystals, and also containing free sulphuric acid and as these drops go in containing the crystals, and the free sulphuric acid reacts with the ammonia in the gas, it tends, of course, to supersaturate those drops and when you get in the presence of crystals, a degree of supersaturation (662) forms, and the crystals being present the supersaturation is discharged on the crystals. When I say discharged, I do not mean totally discharged, but I

mean it is being discharged on the crystals. In other words, there is an operation set up where ammonia is being fed into the drop at a certain rate, and at that rate or a rate near that, the ammonium sulphate supersaturation being produced is being discharged on the crystals.

Q. In essence then there is happening in the sprayed material from the Otto arrangement by virtue of its spraying a liquid, we call it red here, exactly the same thing that happened in the bath in the early cracker pipe? A. That is right. The Otto saturator does the same two functions as are done in the cracker pipe apparatus, absorption and crystallization.

Q. Both at the same time? A. Occurring simultaneously.

Q. In the same reaction zone. Now did you find any other apparatus in which that took place at one place in a spray; that is where the gas passes through the reaction zone without obstruction and reaction is caused to occur in a spray? A. You mean the reaction accompanied by crystallization?

(663) Q. That's right, the whole cracker pipe reaction takes place without a cracker pipe, in a stream of gas flowing through space without obstruction. A. I don't know of any illustration, any other illustration that I can give where both absorption and crystallization take place at the same time in a spray, and that makes me think that the Otto apparatus is unique in that respect. I think that there are—I know of other places where absorption and crystallization occur in the same unit, in the same zone of a unit, but not in a spray form.

Q. That would be, for example, in the conventional cracker pipe? A. That is one example, and there are others.

Q. On the other hand, where absorption of ammonia is the principal objective, sprays have been used for many years, the crystallization taking place elsewhere. Is that

right? For example, here (indicating). A. I don't know about sprays having been used for many years. In other words, here is a Neumeyer and a Wellstein and a Brunck patent, all about the same, and a Doherty patent, but I can't say that they have been used for a number of years.

Q. At least if you follow the teaching of those (664) old patents, you would spray to obtain the absorption of the ammonia but the crystallization would have to be done some place else? A. At another place. Now I just want to clear one point before I leave it. There is also some crystallization that takes place in another place in the Otto saturator. In other words, as the droplets fall in this Otto saturator, and are reacting—the free sulphuric acid is reacting with the gas and tending to produce a supersaturation, not in the metastable region though, while crystals are present and it is being discharged on those crystals, of course that is true just before the drop hits the pool of liquid at the bottom. As soon as it hits that pool there is no longer any gas, and therefore no further reaction can take place, but at that moment it was slightly supersaturated and was discharging the supersaturation on the crystals that were in the spray and when it gets down in the bath, down below, that supersaturation that exists in there, of course, has to be discharged on the crystals down below.

* * * * *

Q. Having regard to the treatment of the fluid in the Jeremiassen invention—and I will refer to the simplified sketch of that which appears in the upper row of sketches on Plaintiff's Exhibit 32—having regard to the treatment which is given the circulated fluid in Jeremiassen by the pump and other apparatus, the fluid which Jeremiassen states is in a metastable condition, is that fluid agitated in the operation of Jeremiassen's device as he describes it? A. Well, it certainly would be agitated as it goes through the pump.

Q. Now, could you point out in the Jeremiassen patent any other differences than the quality of the circulated solution between Jeremiassen's process and apparatus and the process and apparatus of the Otto spray saturator patent in suit? A. Yes, there are differences in the way that he makes his contact with the absorbing solution and the gas, because (673) he wants to get what he calls a falling rain or veil or curtain—I think that "veil" is the word that he uses—but that is a different type of contact situation from what you get in a spray, and he says that he does not want a spray and he says—or in effect he says that—and he does not want a packed tower either, because either one of those types of operation in his thinking, as I read it, would defeat what he is trying to do. Now, the reason that he does not want a packed tower, I think I indicated yesterday, is that if he had a packed tower and this saturated solution were held up in that tower for any length of time locally that saturation would become a supersaturated condition on beyond what he wanted, and would tend to produce crystals.

If you take a droplet or a little piece of chalk and drop it through the height that he might have in his tower, ten or fifteen feet, or his chamber, it would obviously fall in something like a second or less, and if you took the same droplet or a quantity of solution and let it flow down through a packed tower the time would be very much longer, and in a packed tower of any sizable height, five, ten or fifteen feet, the time could run into minutes from the time the material is introduced at the top (674) to the time that it flows out the bottom, and he does not want it to be held up there any great length of time.

He does not want a spray either, because droplets of a spray would get, he says, on the walls of his chamber and tend to become supersaturated to the point where crystal formation would take place and he wants to avoid these

incrustations, and he carefully designs his apparatus so that the boundary—I was going to say side walls; they weren't exactly walls in the sense that they are a total enclosure—the boundary of the space between the gas inlet passage and the inside of the chamber where he has this falling rain slope, and the gas is always going inwards, so that he tries as hard as possible to keep drops away from the walls, if you can call it that, of his gas chamber. In other words, he wants to avoid a spray effect, and he does not want a packed tower either.

Q. How about comparing the structure called for in the Otto patent with respect to the contact achieved between gas and liquid and between the wetting of the walls? A. Well, in all cases in the Otto design he has a very heavy spray in his chamber and also a heavy spray going against the walls of the chamber.

Q. In other words, instead of keeping the walls dry he keeps them extra wet, is that right? (675) A. Yes.

Q. And with respect to the place where the release of supersaturation is to take place in the crystal-forming portions of the two apparatuses, is there a difference between the two, Jeremiassen and Otto? A. Well, in the Otto process, with plenty of crystals in the circulating stream, the supersaturation which is being produced by reaction of the gas on that solution is being discharged partly in the gas space as the spray falls through—

Q. That space right here underneath the sprays? A. Yes, sir. Partly in the connecting pipe between the absorbing section and what is denoted in the Otto patent as a crystallizer—

Q. That's this tank here to which the lower connecting pipe discharges, is that right? A. Yes. And the balance of the supersaturation over there in the crystallizer. In other words, in the Otto process the supersaturation is being discharged en route to the final tank over there.

Q. What does Jeremiassen say about the discharge of his supersaturation? A. His process, which is based on the Isaachsen idea, the supersaturation which he is getting in the falling (676) spray, he does not disclose any discharge of that in the spray and he does not disclose any discharge of that en route to the place where the crystals are, but he does say that the supersaturation is being discharged, or the solution is being desupersaturated, down there where the crystals are.

Q. That is to say, in the bottom of the part marked red on your Exhibit 32? A. Which is a point somewhat remote in the sense that there is a connecting passage—

Q. That's this downcomer here on this drawing, is that right? A. Yes, a point remote where the supersaturation is produced.

* * * * *

(678) Q. Now, Professor, I show you Defendants' Exhibit 16, which is an illustration of the spray saturator owned by the defendant Wheeling Steel Corporation and operating at Follansbee, West Virginia. Can you tell us whether, from that drawing and from any other studies you may have made of the construction and operation of that saturator—first, whether the apparatus and operation for affecting the contact between the gas and the liquid therein follows the teaching of the Jeremiassen patent, that is, in the form of a veil, or whether it corresponds to what you have characterized as the spray method of the Otto disclosure? A. Well, this shows diagrammatically a number of sprays, so that I would call this a spray type equipment.

Q. And the sprays are located so that they will do which —avoid the walls as in Jeremiassen, or copiously wet the walls? A. Well, they would certainly wet the walls copiously.

Q. Now, the evidence available here is quite clear that

in the Wheeling saturator the liquid sprayed through the main sprays is a saturated solution of ammonium sulphate (679) containing crystals. Having that in mind would you compare the places where crystallization takes place in the Wheeling saturator with the places where crystallization is described as taking place in the Jeremiassen arrangement and the place where crystallization takes place in the Otto arrangement? A. Well, if this is a solution containing crystals and acid, as soon as the spray is warm, that is, as soon as the liquid leaves the nozzle, the ammonia in the gas with which it is in contact would react with it, and since there are crystals in it the supersaturation which tends to be produced would start to be discharged on those crystals, and would continue in the bath below it until the liquor became desupersaturated completely.

Q. What you have just described is the crystallization that would take place, that does take place, in the Wheeling saturator as you analyzed. That, if I recall correctly, would not be the sequence under which crystallization takes place in the Jeremiassen operation as he describes it, would it? A. In the Jeremiassen as I understand it, the liquor is only supersaturated at one place and then is desupersaturated in another place.

Q. And in the Otto, on the other hand, the crystallization (680) takes place substantially in the same manner and in the same places as in the Wheeling, is that right? A. Yes, sir.

Q. Now, in the Jeremiassen patent the circulation is described as in the metastable range throughout, is it not?

The Witness: You mean the circulation of the liquor leaving the—

Q. The liquor leaving the pump and on its way back to the crystallization space is described by Jeremiassen, I believe, as a circulation in which the metastable limits

are never exceeded, is that right? A. I think I would have to refer to the Jeremiassen to see whether that is the case.

Q. Please do. A. Well, he doesn't describe it exactly that way.

Q. How does he describe it? A. He says, "An important characteristic feature of the process which is the object of the invention consists therein that the solution to be concentrated by contact with the gas is introduced into the gas space in an approximately saturated condition." That is line 14 on column 2, and then farther down, line 33, he says, "Referring to Figure 1, the numeral 1 designates the pump forcing (681) approximately saturated solution up into a container 2."

Q. So that the portion which is circulated through Jeremiassen's is described as being saturated and as coming down without ever exceeding the metastable limit, is that right? A. In the coming down part it does not exceed the metastable limit.

Q. Would it, therefore, in the coming down part contain any crystals? A. Not the way I understand metastable solutions.

Q. How does that compare with the circulation in the Wheeling saturator as shown on Defendants' Exhibit 16? A. Well, this circulation here, as I understand it, contains crystals and therefore it is a seeded solution, as in the Otto process.

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Cross Examination by Mr. Brown:

* * * * *

(686) Q. I asked you about your personal knowledge as a teacher and professor of chemical engineering, Professor, and on that basis will you say whether or not you yourself personally knew before 1946 of the availability of

the process of spraying a liquid through a gas to cause some chemical reaction or to cause any effect or interaction between the liquid and the gas? A. I knew of the availability of sprays to cause such reactions.

Q. And before 1946?

The Witness: Before 1946?

Q. Yes, sir. A. Yes, sir.

(687) Q. Wouldn't you say that any qualified chemical engineer in or prior to 1945 had such similar knowledge? A. Well, if you mean by "any", any possible one, I would say yes, but if by "any" you mean all qualified chemical engineers I would say definitely no.

Q. Well, anyhow, a fair proportion of them had such knowledge—you agree with that, I assume? A. They had such knowledge of spray devices to get contact with liquid and gas.

Q. Yes. A. Yes.

Q. Did you mean to tell the Court that in your research and study leading up to what you called the evolution of ammonium saturators, that you had found no references in the published art or patented article of a sulphuric acid solution being passed in finely divided form through ammonia gas for the purpose of removing ammonia prior to the Otto spray patent in suit? A. I found such a reference, yes.

Q. You had found such reference? A. Yes, sir.

Q. Well, I must have overlooked it. What was that reference? Did you refer to it in your direct testimony? A. No, I have not referred to it.

(688) Q. Well, would you mind telling me what it is now? A. No, sir. It is the Tiddy patent 1,997,757 filed in August, 1931, and granted in 1935.

Q. What does that patent teach you as a chemical en-

gineer and what, in your opinion, does it show as to projecting sprayed sulphuric acid solution into a space containing coke oven gas or other gas to be treated by the sprayed liquid? A. Well, it taught me a lot, because I knew something about that development and I knew that Mr. Tiddy had in mind the fact that a spray from a spray nozzle to do this job might not work on the basis of some other experience that he had had and that he devised a particular kind of spray equipment to do this job, which I distinguish from a spray tower type of equipment.

Q. Well, so far as the interaction of the sprayed sulphuric acid solution into coke oven gas, the fine particles thrown up by the rollers in the Tiddy patent would interact with the coke oven gas and remove the ammonia in the form of ammonium sulphate, would they not? A. That's correct.

Q. So that there, as you understand it, before the date claimed for his invention of the spray patent by Dr. Otto, which, for your information, I will tell you on the record (689) was in May or later of 1946, so that this Tiddy patent, as you interpret it, showed that a prior inventor had proposed to remove ammonia from coke oven gas by spraying, or call it what you will, projecting fine particles of sulphuric acid solution through the gas? A. Mr. Tiddy very definitely did propose that.

Q. Have you the patent before you? A. I can get it.

Mr. Brown: May I hand a copy to the Court? And it may facilitate things if we now mark it as Defendants' Exhibit 37.

Q. Professor, this Tiddy patent describes spraying the sulphuric acid through the coke oven gas. You have agreed with that? A. Yes, sir.

Q. Will you also agree that this sprayed material was

picked up out of the mother liquor bath which contained ammonium sulphate crystals? A. We haven't agreed to it yet. I can agree to it now.

Q. Sir? A. You asked if we had agreed to it.

Q. Yes. (690) A. I can agree to it now.

Q. You agree to it now? A. That this bath down here contains ammonium sulphate and ammonium sulphate crystals.

Q. And will you agree that the material picked up and sprayed through the gas contains ammonium sulphate crystals? A. I think that would be the case, because while the bath is very deep there are undoubtedly crystals at the top of the bath. I am not too sure about that, though, Mr. Brown, because I don't know exactly what this condition would be at the top of the bath, but I think there are crystals in that spray liquor.

* * * * *

Q. I was quoting that sentence, "The sprayed liquor containing ammonium sulphate, both dissolved and crystalline," that refers and is a statement by Tiddy that his spray (691) did contain ammonium sulphate crystals? A. Yes.

Q. You agree to that? A. Yes, I agree.

Q. Will you also agree that in the apparatus, which for the purposes of my question is illustrated in the upper part of Figure 2 at the right of the drawing, the space between the roller and the casing in which the gas is subjected to spray is unobstructed? A. Yes.

Q. In other words, sulphuric acid solution containing ammonium sulphate crystals is sprayed through coke oven gas in an unobstructed space, as I understand it? A. Yes, I would agree to that.

Q. Now, Professor, I believe you only referred in your development of the evolution of ammonium saturators to publications. You didn't go out into the commercial art

at all, as I understand it. A. I don't know what you mean by commercial art, as distinguished from publications.

Q. Well, I mean saturators as they were built and used and operated in the field. A. Well, I had seen some during the course of years.

Q. Well, sir, I think we are misunderstanding each (692) other. I mean your testimony was based upon publications rather than upon any commercial installations. A. That is correct.

Q. Did you know about the Underwood saturator at Bethlehem which has been proved here while you were sitting in the court room, did you know about that before you heard of it in this court room or in your preparation of this case? A. Not before that.

Q. But assuming that that Underwood-Bethlehem installation operated as has been described by Mr. Helm and Mr. Stout, that also would be an instance in which sulphuric acid solution containing ammonium sulphate crystals was sprayed through an unobstructed space through which coke oven gas was passing, would it not? A. I would not agree to that.

(693) Q. Well, let me try something else. Now, the chart Defendants' Exhibit 24 shows the complete cycle of operation that is described in the Jeremiassen patent, does it not? A. Yes, sir.

Q. And your diagram shows the crystallizing chamber and a part of the main leading to it and the part of the main leading from it to the spray chamber? A. Yes, sir.

Q. Now, Professor, if there were entrained crystals in the mother liquor carried from the crystallizing chamber to the riser pipe 1, and the horizontal portion and then projected through holes in the end of the pipe down through the chamber, through which the coke oven gas would pass, contained entrained crystals, would you believe that that showed an instance in which coke oven

gas was treated with sprayed sulphuric acid solution containing entrained crystals? A. If there were entrained crystals in that, I would say the gas was contacted with a liquid containing crystals and sulphuric acid. I still would not call the rain or veil there a spray.

Q. Well, you will agree that the liquor is projected out of the main through holes in what might be called (694) streams, and that the patent says that the liquor is broken up into drops? A. Yes, sir.

Q. And the only distinction you are making, as I understand it, is that the liquor is not projected out through a spray nozzle. Is that right? A. Well, the objection I am making is that Jeremiassen did not want a spray and he designed his equipment in terms of a veil or a curtain specifically, and disclaimed a spray.

Q. May I interrupt you? I beg your pardon for doing it, but I would like to have you answer my question rather than argue about what Jeremiassen wanted. I am talking about what Jeremiassen shows now. A. He did not show a spray.

Q. How do you define a spray, Professor? What does it mean to you as a chemical engineer? That is all I am asking you. How would you define a spray? A. A spray is, of course, that which you would get coming out of a spray nozzle.

Q. Coming out of what? A. A spray nozzle. I would say it is a very random distribution of droplets through the gas space. This is not that.

(695) Q. Well, isn't the difference between projecting liquid in finely divided forms through a gas, through a spray nozzle, and allowing it to pass through a gas from any other source in finely divided form, merely a matter of terminology? A. The answer to that, I guess, is yes, depending upon who makes the terminology.

Q. Well, even in your terminology? A. In my termin-

ology, projecting a liquid, or letting a liquid fall through a space is a different thing from spraying it in random direction.

Q. The functional effect of the liquid passing through the gas is dependent upon the size of the particles of liquid, and the number of particles, is it not? A. That is one of its functions.

Q. Well, that is the function we are talking about (696) in these spray type saturators, isn't it? That is, you want to get contact of liquid surface with the gas? A. We have talked about other functions.

Q. Let's talk about this one. That is the main function, isn't it? A. Certainly from that aspect, that is a function, to sub-divide the liquid so that it gets better contact with the gas.

Q. And in this particular function—if you wish to confine yourself to it—does it make any difference whether the liquid introduced to the gas chamber is through a spray nozzle or through a strainer or through a perforated space, or what-not, provided the same amount of liquid in finely divided form is passed through the gas? A. Well, in a very general sense, that is so, but there are differences between drops flowing one way and drops flowing another. In other words, drops from a spray might have great velocity through a gas, and velocity is also a factor, as well as area, so if I had a drop which is falling in a vertical direction, I could get one value in terms of efficiency, and if I got drops which go from sprays, I could get different values for efficiency, but they both produce area.

(697) Q. And they both would absorb ammonia from coke oven gas if the same quantity of liquid was passed through, which you call a spray nozzle, as distinguished from being passed through some other means of injecting the liquid into the gas? A. They both would absorb ammonia from the gas, regardless of the volume.

Q. I didn't intend to start any fencing match with you, Professor. I think that your answers come out the same way in spite of the fact that they seem to be somewhat argumentative. Have you any doubt that the introduction of liquid as shown in the Jeremiassen patent would be sufficient to remove the ammonia from the coke oven gas? A. No doubt.

Q. And in order to remove ammonia from coke oven gas you have to produce ammonium sulphate crystals, do you not? A. No.

Q. How else do you take the ammonia out of the liquor after the liquor has absorbed the ammonia in one form or another? A. If you absorb ammonia in a liquor, you can distill it out.

(698) Q. Do you know, or have you ever heard, of any commercial installation, or any saturator, that you know about or heard about, which doesn't remove the ammonia from coke oven gas in the form of ammonium sulphate crystals? A. Well, ammonia can be removed from coke oven gas in the form of ammonia liquor, and I have heard of that.

Q. Does Otto do that? A. No, sir.

Q. Do any of the references that you have—any of the saturators you have talked about, do that? A. No, sir.

Q. All of them produce ammonium sulphate crystals, if I understand correctly? A. Yes, all of these processes that remove the ammonia produce ammonium sulphate crystals.

Q. And Jeremiassen, will you agree, proposed to take the ammonia out in the form of ammonium sulphate crystals? A. Yes.

Q. And will you agree that he proposed to have the crystals formed, in part at least, in the crystal suspension chamber marked 11 in the chart? (699) A. He proposed to have them formed there.

Q. And the liquor that he injected through the coke oven gas was carried directly from this crystallizing chamber through the riser 1 and showered down through holes in the end of that pipe? A. Yes.

Q. Now if the crystals were formed in this chamber 11, crystallizing chamber 11, and you drew liquor from that chamber through the pipe that is shown, would you not expect to have entrained crystals in that liquor? A. You mean what I would do, or what Jeremiassen disclosed?

Q. I am talking about you now. I am asking you the question what you would expect. A. If I designed the process according to his disclosure, I would have to take that liquor out of there without crystals.

Q. Take the apparatus you are looking at. A. Yes, the crystal stopped at a certain point in this crystallizer and then it is clear red from that point on, as you have shown it.

Q. Is it your statement that in your opinion the operation of the Jeremiassen process in cycle as illustrated on the chart before you, which is taken (700) directly from the patent, that there would be no crystals in the crystallizing chamber at the point where liquor is drawn off to be returned to the spray chamber? A. My opinion, from looking at that chart and reading the Jeremiassen patent, is that there are no crystals in that liquor.

Q. I wish you would answer without reading the Jeremiassen patent. A. You asked me that.

Q. I am asking you as a chemical engineer if you, in your opinion, if the plant built, constructed and operated as shown on the figure before you, whether there would be no crystals in the stream drawn off from the crystallizing chamber and returned to the—what I call the spray chamber, and you can call shower chamber. A. It would have to be properly designed to do that, and if it were, you could get a solution with no crystals.

Q. Take the design in front of you. As a chemical engineer, do you wish to say in your opinion there would be no entrained crystals in the returned flow? A. The design which is in front of me shows a (701) limit of crystals before you get up to the suction of that pump.

Q. You wish to go on record as saying, in your opinion, there would be no entrained crystals in the operation of the cycle and apparatus that is illustrated in Defendants' Exhibit 24? Just say yes or no. I want to know if you want to put your professional reputation on that. A. I think a plant could be designed with no crystals.

Q. I didn't ask you that. I asked if taking the plant that is before you and operating it as it is illustrated and described—that is illustrated—on the chart before you, is it your statement as a chemical engineer that in your opinion there would be no entrained crystals returned to the spray chamber in the operation of that cycle? A. I think there should be no crystals in that line.

Q. You say you think there should be none? A. That's correct.

Q. You mean that there would be none? A. I have never seen one of these plants, sir. I have never built one.

(702) Q. In your opinion as a matter of fact—is it your opinion that as a matter of fact there would not be any crystals entrained in the return of the mother liquor to the spray chamber? A. I can't answer that question.

Q. Now a while ago you said that you would not agree that in the operation of the Underwood Bethlehem saturator as described by Mr. Stout and Mr. Helm, that there would be entrained crystals in the sulphuric acid solution projected through the gas chamber. A. I did not say that.

Q. Well then, let me ask you that question. What is your answer to the question as to whether there would be, under the condition stated in my preceding question? A. I think there are crystals in that spray and it has been testified that there were.

Q. And that would be a case not in the printed art, but in the practical art, where sulphuric acid solution containing entrained crystals were projected in liquor, finely divided liquor, through an unobstructed space through which coke oven gas was passed? A. No, sir.

Q. Why do you say no to that? A. Because I use the term unobstructed space as (703) it is used by chemical engineers in the art, by chemical engineers in the Koppers Company, and elsewhere, as to describe an unobstructed space as the space that exists from the point of inlet to the point of outlet, and in any cracker pipe installation, and that is what Bethlehem is, there is an obstruction there. The spray thing that Underwood put in was an experiment and an incidental thing, and—

Q. Now, Professor, let's don't harrangue each other. Will you answer my question? I will ask you another one if you want me to. A. The question has been answered no.

Q. As I understand your answer, it is no because you say there was an obstructed space. A. Yes, sir.

Q. Now except for the obstruction which you say was caused by the cracker pipe, would there be an instance of spraying sulphuric acid solution containing entrained crystals through coke oven gas for the purpose of removing ammonia? A. In that particular space, yes. Excuse me a minute. You say for the purpose of removing ammonia. I don't think much ammonia would be removed by that spray.

Q. Well, you think some would be removed by that (704) spray, do you not? A. Well, some in the sense that there was part of the last one, two, two and a half percent.

Q. That is important, is it not? A. That is vitally important to get it all out.

Q. I think we are back where we started, are we not? A. I am not back there, because I do not think that the

saturator that was in existence at Bethlehem is absorption of ammonia from the gas in an unobstructed space, because I have to define that obstructed space, or unobstructed space, in terms used in the art, which means from the point of inlet to the point of outlet.

Q. Do you know what Doctor [Otto] himself has given as the definition of unobstructed in his patent, as he understood it? A. Do I know his definition? I can't recall that.

Q. I think I can tell you he said it mean, to him an empty space. A. Yes.

Q. And he meant space which didn't have pipes passing through it and sprays, and so forth, which might become (705) encrusted with ammonium sulphate crystals. That is as I understand what he meant. Do you accept that proposition? A. I accept that in part, yes, sir.

Q. What part do you not accept? A. Well, I mean I accept that as part of the picture of an unobstructed space. There is another part; say for example it did not contain packing or baffles or cracker pipes.

Q. I just want to make sure we do understand a point which I think is important. That your only distinction from the spraying of a sulphuric acid solution containing entrained crystals through coke oven gas as exemplified in the Underwood, Bethlehem apparatus, which has been proved in your presence, is that you say there was an obstructed space through which the gas and liquid passed. Is that right? A. Through which the gas passed.

Q. Through which the gas passed? A. Yes, sir.

Q. We are agreed on that then? A. Yes, sir.

Q. Now, do you remember, Professor, the Otto bell-mouth type saturator that was put in at Wheeling and which (706) is charged to infringe one of the patents in suit? A. Yes.

* * * * *

Q. And, Professor, I will show you a copy of that patent and ask you to look at Fig. 6 on page 2 of the patent. A. Yes, sir.

Q. At the right-hand side of the Fig. 6 you see kind of a nebulous figure marked B-4, which the text describes as a spray forced up into the gas chamber by gas passing through the bath and then up into the chamber. Is that your understanding? A. Yes, sir.

Q. And the patent also states that this spray forced up by the gas through the liquid bath is sufficient to wash the walls of the tank to prevent salting up. Remember that? A. I don't remember that, but I will take your word for it.

Q. Well, take my word for it, and assuming that that (707) is so, is not that an instance in which a spray is forced through coke oven gas, the spray consisting of a liquid containing entrained crystals? A. This is practically the same as you would find in all cracker pipes.

Q. Isn't it true then that in view of the way you understand it, that all cracker pipes involve the production of a spray passing through the coke oven gas, that spray consisting of sulphuric acid solution containing entrained crystals? A. There is a spray from all cracker pipes and in all apparatus of this kind which gets into the gas. I do not quite agree that the spray passes through the space. That means something a little different to me.

Q. Well, it passes upward into the space? A. Yes, sir, and upward into the gas.

Q. If it was sufficient to wash the walls of the tank, it would pretty well pass through the space, would it not, or at least a large part of the space? A. Oh, yes, it would have to pass from the point of egress of the solution over to the wall.

Q. Yes, and up the wall too, if you want to keep them clean, wouldn't it? A. Yes, up the wall for quite a distance.

(708) Q. Do you question that that spray would carry entrained ammonium sulphate crystals? A. No, sir.

Q. You gave the court a somewhat—what seemed to me a somewhat theatrical exhibition yesterday on your illustration of the metastable solution. Was that made with the kind of mother liquor involved in the saturator we are talking about? A. No, sir.

Q. Not made with any material that is involved in any saturators we are talking about, was it? A. The only material that was involved there was water, that is used in saturators.

Q. Professor, you went at great length into this metastable matter, and I frankly admit your superior knowledge as far as I am concerned, so I am not going back into it with you, but as I understand your exposition of this matter, you did not cite any authority. By that I mean recognized treatises, text books, and so forth. You know what I mean by that. A. Yes, sir.

Q. But merely gave your own definition and exposition of the matter you discussed. Is that right? A. I did not cite an authority but I got my (709) definition from an authority.

Q. Which authority you did not cite? A. I did not cite it.

Q. I want to ask one other question on this and that is all. If I understood you correctly, you said it was impossible to have a metastable solution, saturated solution, in the presence of crystals. A. That is my understanding of it, that metastable solution means no crystals.

Q. That is what you meant to tell the court yesterday? A. That is correct.

Q. That is what I understood you meant to say. A. That is what I meant to say.

Q. If I am wrong about it, I would like you to correct it now. Now, Professor, you purported to show the court

the evolution of the saturator art, and particularly, among other things, you showed the evolution of the cracker pipe type saturator. Is that right? A. Well, I hardly think I showed the evolution of the cracker pipe. I showed a Brunck patent and one other Becker patent, but there have been a lot of other developments.

(710) Q. You didn't show the court the cracker pipe Otto saturator which is described in the patent in suit here, did you? A. No, because to me that is another illustration of the cracker pipe art.

Q. There has been a gradual evolution and improvement even in the old cracker pipe? A. Oh, yes.

Q. You observed that as you went along? A. Yes, sir.

Q. And that evolution had progressively lowered the differential between the two sides of a cracker pipe saturator, did it not? A. Yes, sir, they were all working in that direction.

Q. So just before 1946, the cracker pipe art had reduced the differential down to a few inches. Is that right? A. There have been a number of developments in the cracker pipe art which reduced the differential.

Q. Very materially? A. Yes, materially.

Q. And that was progressive? A. Oh, yes.

Q. Now, Professor, I think you would agree as a (711) teacher of chemical engineering that there is in nearly all arts a progessive development and improvement of apparatus as they go along. Isn't that right, in your experience? A. There is until that art ends.

Q. Does it ever end? A. Yes, it ends sometimes when another type of development comes along and makes it obsolete, then it ends.

Q. In other words, until something supersedes it. A. That is correct.

Q. And then maybe it will be completely discontinued? A. Correct.

Q. But usually there is a progressive improvement as you go along— A. Yes, sir.

Q. —throughout the life of any apparatus or process. Is that right, in general? A. I would think so.

Q. Professor, if I understood you correctly, you referred to the diagram at the lower right-hand corner of this chart, Plaintiff's Exhibit 32, and you were asked what the X across the middle of the tank was, and (712) you said that was the conventional way of indicating a packed tower. Is that right? A. Yes, sir.

Q. That is your understanding? A. Yes, sir. I probably should say that is a conventional way. There are other ways.

Q. It is a standard way of doing it? A. Yes, sir, quite standard.

Q. Yesterday in discussing the Doherty patent, and a simplified drawing of which appears on Defendants' Exhibit 19, you stated that the red liquor shown in the splotches across the bottom of the spray tower would clog up with crystals and become inoperative. Is that right, in your opinion? A. No, that is not quite right.

Q. Then correct me, please. I am not trying to misquote you. I want to understand. A. I said if that became saturated or supersaturated, so that crystals were forming in there, it would become plugged up.

Q. If that acted as a reaction chamber for producing crystals, it might clog up? A. Not only that, but if the liquor coming down to that were received so that it contained crystals, (713) it would plug up.

Q. Now would you say that if it had been desirable to remove crystals from the pool of liquor shown in red at the bottom of this, that would have been beyond the skill of chemical engineers to devise means of taking crystals out? A. I do not understand that, because that is not what Doherty talks about at all.

Q. I am asking you now from your professorial ability and as a chemical engineer. A. Could I make something out of this?

Q. No, could you get the crystals out of the bottom if it was desirable to remove them, from the basin which is shown by the red color? A. I do not think I could.

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(714) Q. Professor, in these saturators which do you regard as most important, the removal of ammonia or the production of ammonium sulphate crystals? A. Well, I think they are equally important.

Q. Well, will you accept the suggestion that it is necessary to remove the ammonia, and that it is (715) desirable to produce ammonium crystals, because they form a salable by-product. Will you agree with that? A. Yes, sir.

Q. Professor, would you regard the word whirling as properly applicable to a liquid after it has left a nozzle through which it is projected? A. No, sir.

Q. It is a misnomer, would you say, or, as you said yesterday, incongruous or something like that? A. In connection with your question, yes.

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Re-direct Examination by Mr. Mofton:

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(753) Q. Now, having reference to Defendants' Exhibit 24 a moment, is this, in the first place, a working drawing? A. Well, I do not understand it to be that. I understand it to be just a diagram.

Q. And is there given on this drawing, including not only the drawing but any legends that may appear on there, whatever their source, is there sufficient data given on there

with respect to dimensions, levels, pressures, and whatever other things you might require, from which you could make an engineering analysis of the structure? A. Well, there is nothing on the drawing and there (754) is nothing in the patent, which is very short and very meager, when it comes to data.

Q. So that before you could determine the details of flows and the like that would be encountered when building such a structure, you would necessarily have to make—either make certain assumptions or designs, which are not indicated on the drawing. Is that right? A. That is correct.

Q. And I remember you answered Mr. Brown—he asked you as an engineer if you couldn't tell him something about this and you said you could not answer that question. Have I outlined some of the reasons, at least, why you couldn't answer that question? A. That is right.

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(756) Q. In addition to removing the ammonia by the process of passing through a saturated or supersaturated solution of ammonium sulphate containing crystals and sulphuric acid, are there other processes for removing ammonia? A. Scrubbing the gas with water.

Q. And in addition to scrubbing it with water and saturated acid, I take it you can take a middle ground and scrub it with unsaturated acid, as you have shown here. A. Yes.

Q. Is that right? A. Yes.

Q. Now suppose that you didn't—that you scrubbed it with water or with unsaturated acid as shown here, and you didn't fix the ammonia in the form of ammonia sulphate crystals, what could you do with the liquor that contained the ammonia? A. You would generally take the ammonia liquor to another place and distill it and recover the ammonia (757) after it has been removed from the liquor, usually as sulphate.

Q. You could not just wash it with water and run the water off on the ground or into a convenient stream, could you? A. Well, you could, but I don't think you would be allowed to do it.

Q. It would make a severe public nuisance, would it not? A. That's right.

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(758) Q. Now, Professor, you might, if you will, tell me, and tell the court, having in mind not only the various prior art devices which you have selected for Defendants' 32, but also any others that have come to your attention during the course of this trial, and in particular the Tiddy device that was introduced by Mr. Brown on your cross-examination, the one with the cylindrical rolls producing spray, will you tell us in what respect the Otto process and apparatus differs from what went before it, as you see it. What is the significant difference? A. As I was making this analysis, and making these sketches leading up to the formation of this chart, I put below that center line the processes where unsaturated solutions of sulphuric acid are used, and I distinguish the Otto process from those because of what I would call that scientific difference, which certainly could be, in my thinking, a basis for a patentable difference. The Otto process had to be (759) compared with the ones above the line, which are the cracker pipe art and the Jeremiassen. As far as the final result is concerned, the Otto process produces the same result as the cracker pipe technique, in that the ammonia is absorbed in the acid and also crystallization is taking place right in the same zone.

There have been other developments of the cracker pipe, particularly these ones that have been discussed with sprays, like the Bethlehem and Monessen, and possibly one or two others, and I think it has been testified to here that basically the reaction is a fairly simple one, fairly easy to

carry out, and that even if the seal on the cracker pipe is lowered to zero, a very high recovery is still obtained, 97½% was mentioned in one case.

Except for variations and capacities from plant to plant and throughout per square foot and that sort of thing, I presume that the order of magnitude of ammonia recovery in a cracker pipe operated that way, any one would be very high.

Now these sprays which have been cited in the past, as far as I can see, were incidental, and the most they could do would be to go after the last two and a half or so percent, which of course is important, but from a (760) standpoint of the engineering job done, the absorption job, they were incidental.

Now I think the thing that Doctor Otto did was to sweep all of that stuff aside and say we will do the whole job in the form of a spray, and to me that is a very distinct advancement in the art.

And as far as Jeremiassen is concerned, which is the other one I have up there, I think there are differences. In the first place, Jeremiassen does not want a spray. He wants a rain. He doesn't want a spray because he says he doesn't want it to hit the side.

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(761) A. (continuing) The difference between Otto and Jeremiassen, as I said, is that Jeremiassen wants this rain and Otto uses a spray, which I think engineeringly is a significant thing. The Otto process discloses that the circulation consists of a solution of sulphate, sulphuric acid, including a suspension of crystals. The Jeremiassen process does not disclose crystals in circulation. That is another distinction between the two. The third thing is that Otto, as I said, accomplishes absorption and crystallization as does a cracker pipe in the same zone, the crystallization is continued as the solution from the bottom

goes over towards his so-called crystallizer, whereas the crystallization in the Jeremiassen patent is stated to take place at a point remote from where the absorption takes place.

(762) Now on the Tiddy patent, which is not shown on the chart, I recognize from the standpoint of engineering that the method of generating the spray is a different one, and I think that Mr. Tiddy had in mind in 1931 that sprays would not have worked, because he knew of another case where sprays from spray nozzles did not work, and he knew from that case also that spray rolls did work, and on the basis of that engineering device, I think there is a difference between the two.

Q. Now, Professor, I just want to clear up what may seem a little difficult for you to testify about, was Mr. Tiddy at any time an employee of an organization for which you were also an employee? A. Well, I think the answer to that is no. I was employed by the Barret Division and he was employed by the Semet Engineering Corporation. I think it was at that time, and each exercised authority over its own employees. They were both controlled by Allied Chemical and Dye.

Q. Did you ever work on projects involving roll type spray apparatus that you know Mr. Tiddy also worked on? A. Well, I worked on spray roll types of apparatus and I know he was interested in those, and I had to consult (763) with him on the use of that type of apparatus for ammonia recovery.

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Re-cross Examination by Mr. Brown:

(769) Q. If you took this metastable gadget that you have talked so much about out of Jeremiassen and just considered the Jeremiassen arrangement as being one in which

mother liquor containing entrained crystals was pumped from the crystallizing chamber to the top of the gas chamber and then sprayed down through the gas, wouldn't you have practically the same thing you say Doctor Otto (770) covers in his patent? A. Not doing it that way, I would not have. The difference would be that Jeremiassen has a rain or veil which I recognize as something different engineeringly, and he does not want a spray and he does not want it to hit the side walls.

Q. Don't talk about what he doesn't want to do, but talk about what he does. Call it a rain or a spray, we agreed this morning that that breaks the liquid up into small drops and causes it to fall down through the gas chamber, and I think you agreed this morning that that would be sufficient to take the ammonia gas out of the coke oven gas, didn't you? A. I did not agree that it broke it up into small drops. I did agree that it would take the ammonia out of the coke oven gas.

Q. All right, then, whether you call it a spray or a shower or a veil, or what-not, the liquid falls freely down through the gas and is sufficient to take ammonia out of the gas. Do we agree on that? A. Yes.

Q. Doesn't that have substantially all you have attributed to Doctor Otto, except that you say Doctor Otto has entrained crystals in the liquid which he (771) showers or sprays or pours, or carries by back for all we care, down through the gas chamber, sufficiently to take out the ammonia from the gas chamber. Isn't that about the only difference you can point out in the broad general practicing of the two processes? A. Well, Doctor Otto, I don't believe, said that he showered or sprayed or poured it down there. He said that he sprayed that.

Q. All right, he said he sprayed it. If we should call the Jeremiassen shower, as you call it, a spray, then what would be left to be distinguishingly different in the way

the two processes are carried out in Jeremiassen and Otto? A. If you should call it a spray?

Q. Yes. Permit me to take the liberty of calling it a spray. A. Well, I think the thing that is then left is the fact that Doctor Otto discloses circulation of mother liquor plus crystals and Jeremiassen does not disclose any crystals in that circulating line.

Q. Then we come down to my question. The only difference would be, that you see, Doctor Otto included crystals in his spray line and Jeremiassen did not. Would that be the difference you find? (772) A. On the supposition that one of the differences is to be voided, then that is a remaining difference.

Q. And the only remaining difference, isn't it, in essence? A. No, there is one other remaining difference. The fact that Jeremiassen produces his absorption in one place and deposits the supersaturation made because of absorption in another place.

Q. Well, both of them do the crystallizing, the formation of crystals, in substance, in the crystallizing chamber, do they not? A. Jeremiassen discloses that that is where he does it. Doctor Otto discloses that it takes place in the transmission line over there as well as in the chamber.

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Re-direct Examination by Mr. Morton:

(776) Q. Professor, I show you Defendants' Exhibit 18, one of the patents contained in there, a patent granted to Sperr, or more accurately, to the Koppers Company as assignee of Sperr, September 26th, 1933. Do you find in that patent a mechanism for producing a spray which is the same as the spray producing mechanism of this Tiddy patent which became Defendants' Exhibit 37 this morning? A. It looks substantially the same to me. He has

five spray rolls in here instead of Tiddy having two, but otherwise it was an apparatus which looked about the same.

Q. How many did Tiddy have? Two? A. Yes, two.

Q. Take a look at the patent. A. I am sorry. Tiddy patent does have three and (777) Sperr has five.

Q. But essentially those rolls are performing the same function in the same way, as far as they are described in those two patents, Tiddy and Sperr. Is that right? A. Well, I would assume so, although I have never seen the Sperr patent before.

Q. And that method of producing spray is one with which you are familiar as far as your engineering practice and training, is it not? A. Yes, I had some experience with it.

Q. Is that method of producing a spray interchangeable in results in all cases and places with other methods of producing spray which have been described here during the trial? A. No. It is a different device engineeringly and in the situations with which I had some familiarity spray nozzles wouldn't work, but this type of device did work, so you can't say that one just simply replaces another, in any one of these devices.

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(737) Louis A. Farinet, having been first duly sworn as a witness, thereupon testified as follows:

Direct Examination by Mr. Morton:

Q. Where do you live, Mr. Farinet? A. Hellertown, Pennsylvania.

Q. By whom are you employed? A. Bethlehem Steel Corporation.

Q. How long have you been employed by Bethlehem Steel Corporation? A. Oh, about thirty-six years.

Q. What is your present job at Bethlehem Steel Company? A. General By-Product Foreman.

Q. How long have you been General By-Product Foreman? (738) A. About two and a half years at the present time.

Q. During the period from about, say, 1941 to 1946, what was your job at Bethlehem? A. Turn Foreman.

Q. And as Turn Foreman did you have under your supervision the ammonia saturator apparatus located at that Bethlehem plant? A. Yes, sir.

Q. Did you also have as a fellow employee as Turn Foreman at Bethlehem a Mr. R. Philemon Stout? A. Yes, sir.

Q. How long was he Turn Foreman with you, to the best of your recollection? A. Well, somewhere in '41 I think he took over, and he left in '44.

Q. Do you know how late in '44 he left? A. In the Fall of the year sometime.

Q. In the Fall of the year? A. Yes, sir. I think it was October, if I am not mistaken.

Q. Now, Mr. Farinet, during the period '41-'46, while you were working as one of the turn foremen at the By-Product Plant of the Bethlehem Steel Company, do you recall that any structural or operating changes were made (739) to the saturators there? A. Yes, sir.

Q. Now, Mr. Farinet, that drawing before you has been marked in this case already as Defendants' Exhibit 14 and is intended to be a simplified but nevertheless accurate illustration in a general way of the way those saturators at Bethlehem looked after the changes had been made in them. Do you agree that that's— A. Yes, sir, that's—

Q. Am I correct that at the time these changes were made and for sometime afterward, a Mr. Underwood was superintendent of the By-Product Plant? A. Yes, sir.

Q. And that these changes were made under his direction? A. Yes, sir.

Q. Now, can you tell us about when the first saturator at Bethlehem had this type of change made in it? A. It was in the year '42 we started on it. I think we put two of them in operation the latter part of '42, the Fall of the year sometime.

Q. And subsequent to that did you put any more in operation? A. Later on.

Q. And how many did you eventually get modified as (740) shown in this drawing? A. Four of them.

Q. Can you tell me when the operation of the saturators with this modification was stopped at Bethlehem? A. Yes, that was stopped in '44.

Q. About what time? A. Around June.

Q. And after that date how were the saturators operated? A. The old Koppers system.

Q. That is, no further use was made of those spray pipes? A. No further use; in fact, they were removed.

Q. The spray pipes were physically removed? A. That's right.

Q. During the period when you had the spray pipes in which I understand began with one or two saturators —there were two saturators first in the Fall of '42 and continued through June, '44—did the saturators operate satisfactorily? A. No, they did not.

Q. Would you describe to the Court the difficulties that were encountered, as you remember them, in the operation of these modified saturators? (741) A. Well, the first thing we run into was the nozzle—the sprays cutting the lead out of the saturators.

Q. That was on the first two that were installed? A. That was on the first two, right.

Q. Now, was the cutting done by the lower sprays or the upper? A. The lower.

Q. The lower sprays? A. Yes, sir.

Q. What steps were taken as a result of that difficulty? A. Why, at first we repaired it by a little heavier lead in there, I think, and then we had trouble again and then we brick-lined it.

Q. These drawings now show them with brick linings. That's the way they got to be after you had been running a while? A. That's right.

Q. Did you make any other changes as you went along? A. Well, we did put larger deflectors in them in order to spread out the liquid that was going in there, and probably wouldn't cut as much.

Q. You remember that larger deflectors were, in fact, installed, do you? (742) A. They were installed.

Q. And did you have any operating difficulties with these saturators? A. We had a lot of trouble with our spray lines blocking up.

Q. The spray lines blocked up? A. That's right.

Q. Did you have any trouble with salt deposits inside? A. Salt deposits is what caused our trouble.

Q. Did you have any salt deposits elsewhere than inside? A. Well, yes, we had plenty of salt deposits on top of the sprays, and after we put the bricks in then it just accumulated up on top of the bricks, practically made a cover over it, and the top sprays, I think, is what was causing our salt deposit on top of our sprays.

Q. Did you have any trouble with the circulating pumps? A. We had quite a bit of trouble in there, keeping pumping, due to this blockage in there, and then due to that it would cause a lot of trouble in our packing glands.

Q. Did the introduction of the sprays permit you to lower the pressure difference across the saturators? A. Well, that was the intentions of lowering our (743) differential. We didn't accomplish much on it.

Q. Can you recollect in inches, roughly, how much— A. Oh, we might have dropped about four or five inches.

Q. And did the introduction of these sprays and the operation of the saturators with the sprays in have any effect on the salt production at Bethlehem during this period? A. I'll say it did.

Q. What effect did it have? A. Why, my personal idea is this. Now, when sprays would block up, then you didn't have the spray effect on your gases going through. That would cause a waste in the scrubbing, allow ammonia to escape from the saturator.

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(795) HOMER H. LOWRY, having been first duly sworn as a witness, thereupon testified as follows:

Direct Examination by Mr. Lutz:

Q. Dr. Lowry, will you please give us your residence address? A. 321 Marguerite Avenue, Wilmerding, Pennsylvania.

Q. And will you please give us your technical degrees and where they were obtained? A. I got my Bachelor's degree at Ohio Wesleyan University in 1918; my Master's degree from Princeton University in 1919; my Doctor's degree in Physical Chemistry from Princeton University in 1920.

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Mr. Lutz: Your Honor, Dr. Lowry is written up in a number of reference works, "Who's Who in America", "American Men of Science", and I believe the Court can take judicial knowledge of those works and we will not go more extensively into this man's qualifications. He is an expert in this field.

Q. Dr. Lowry, I believe you have been in the court room and have heard some testimony about the Underwood

spray saturator which is illustrated on a chart, Defendants' Exhibit 14, is that correct? A. That's correct.

Q. Now, can you tell us whether or not you heard about this saturator at the time it was in operation at the plant of Bethlehem Steel Company? A. I did.

Q. And how did you learn about this saturator? (798) A. I learned of it through our association with the Research Department, either through C. W. Stahl or Jack Smyser, I'm not sure which. The Bethlehem Steel Company was a sponsor of the Coal Research Laboratory from 1937, and my associations with them and their developments was close through the period until I left.

Q. In other words, you had regular contacts with these two men that you have mentioned? A. That's correct.

Q. Can you tell us whether or not this information was given to you with any injunction of secrecy? A. It was not.

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(799) Q. I believe you heard Professor Radasch yesterday explain his theory of metastable supersaturation. Do you agree with that theory? A. I do not, as applied to the system that is under consideration.

Q. By "system" you mean the particular chemicals involved? A. Crystallization of salt, such as ammonium sulphate.

Q. And I believe also you heard Mr. Helm give his explanation of metastable supersaturation? A. That is correct.

Q. Just to refresh your recollection, and the Court's, I would like to read Mr. Helm's answer that Mr. Helm gave at page 400 of the transcript. The Court asked Mr. Helm to explain the meaning of supersaturation, and this is Mr. Helm's answer:

"I will try. I might say, your Honor, that the concept of metastable supersaturation, in my opinion,

is a little difficult to describe, but my understanding, as applied to this apparatus and process, is that a solution which is supersaturated (800) within the metastable limit is one which contains more, slightly more, dissolved ammonium sulphate in this instance, than the solution will hold as a stable solution; in other words, it is an unstable condition which will maintain slightly more than the solubility limit of ammonium sulphate for a relatively short time, and within this metastable limit there is a minimum tendency for the production of new ammonium sulphate crystals, which are sometimes referred to as seed crystals, or nuclei, and operating so that the solution as maintained within the metastable range of supersaturation tends to produce the minimum number of new crystals, and to cause the ammonium sulphate which is formed in the reaction to be deposited instead upon the pre-existing crystals which are already present in the system, the net effect of which is to produce larger crystals for a given quantity of ammonium sulphate product than if a larger number of new crystals were permitted to be formed."

Now, I ask you if you agree with Mr. Helm in his (801) statement there of the metastable range of supersaturation. A. I do, and I believe it is fully consistent with other definitions given by experts in the field.

Q. Now will you please explain your reasons for making that answer? A. The reason for making the statement is a review of the literature, some of which I may wish to refer to—not all of it— [the witness here discussed Defts.' Exhibits 38, 39 and 40].

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(807) Q. Now, you have referred to several publications. Can you tell us whether or not, in your study of this ques-

tion of the metastable range of supersaturation, you have studied other reference works? A. I have.

Q. And do I understand that as a result of the study and these articles that you have referred to you conclude that Mr. Helm gave a correct explanation of metastable supersaturation? A. That is correct.

* * * * *

(809) Q. Then can I summarize what you have said on this subject of metastability by asking you whether or not it is possible to have a solution within the metastable range of supersaturation and have crystals present in the solution? A. I definitely believe so, and I believe it is supported by the references which I cited.

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(810) Q. Dr. Lowry, I place before you the chart which is Defendants' Exhibit 24, illustrating Figure 2 of the Jeremiassen patent. In connection with this chart, I believe you heard Mr. Helm state that crystals would be entrained in the liquor circulated to the outlets of the top of the gas chamber. You heard Mr. Helm give that testimony? A. I did.

Q. Can you state whether or not you agree with Mr.
(811) Helm in that regard? A. If one assumes that the proportions shown on the diagram are approximately those that would be used in a plant, and with rates of circulation that would be at all appreciable, it seems to me inconceivable that fine crystals would not be entrained in the liquid which was taken out of the crystallizing tank, which I believe is No. 9, through the pump shown near the top of the surface of the liquor.

* * * * *

Q. I call your attention to the model of the Wheeling saturator, which has been marked Defendants' Exhibit 17.
(813) Are you familiar with the fact that crystals are

present in the liquor which is circulated to the sprays in this saturator? A. I am.

Q. And I believe you heard Mr. Helm testify that there is no particular advantage in having crystals present in this liquor circulated to the sprays. Do you agree with that opinion? A. I do.

Q. Will you please explain your reasons for making that statement? A. Instead of being an advantage, I would say it would be a distinct disadvantage if one is attempting to produce ammonium sulphate crystals in the coarse category, because through the turbulence in the spray nozzles with entrained crystals, those crystals would be broken up and provide many more nuclei for formation of crystals, and the net average size of the crystals produced would be smaller.

Q. And I believe in the Svanoe article, which you just referred to previously, there was some reference there to crystal breakage? A. There was.

Q. Did you witness yesterday the demonstration made by Professor Radasch with these two little bottles of a (814) solution of sodium thiosulphate? A. I did.

Q. In your opinion, would all salts act the way the sodium thiosulphate did in this demonstration? A. Certainly not.

* * * * *

(815) Q. Is it not a fact that the salt produced in an ammonia saturator is ammonium sulphate? A. That's correct.

Q. And how would ammonium sulphate compare with sodium thiosulphate in regard to its reaction in this type of a demonstration? A. I do not believe you could get this type of a (816) demonstration with ammonium sulphate.

Q. Then, to sum up your testimony on this subject, Dr. Lowry, would you tell us whether, in your opinion, it

would be possible for Professor Radasch to have prepared a solution of ammonium sulphate in New York last week and to have brought it into this court and to have made a demonstration similar to the one he made with sodium thiosulphate?

The Witness: Did I understand you to say a solution or did you specify a supersaturated solution.

Q. Well, I meant to say a supersaturated solution. A. I do not believe it could have been done.

(816) Cross Examination by Mr. Morton:

Q. Dr. Lowry, you recognize, of course, that the word "metastable" is an English word used to describe generally a condition not confined to crystallization, don't you? A. I do, and I also recognize that it means not stable; otherwise, it would not be metastable.

Q. And do you accept or reject the general definition of that stage contained in a work by Dr. Glasstone, namely, and I read:

(817) "The term 'metastable' is used to describe a definite equilibrium which is nevertheless not the most stable equilibrium at the given temperature; a metastable system undergoes spontaneous change on the addition of the stable phase."

Do you agree with that statement as a general— A. I question very definitely the definition, because that is a metastable equilibrium, and I question that there is such a metastable equilibrium in the question of crystallization.

Q. You do? Now, I call your attention to the handbook which you yourself referred to, namely, Mr. Perry's handbook, and especially to page 1055, which is one of the

pages which you selected for photostating. There appears on that page a Figure 151— A. That's correct.

Q. (Continuing) A statement describing that figure, which indicates that a solution which is in the supersoluble range—I take that to mean that it is supersaturated in that condition—will, if a crystal is added, drop to the normal solubility curve—which I take to mean the saturated level. You think that that handbook is wrong in that part, I take it? (818) A. I am not sure it is necessary that a crystal be added—it's a question of probability, as stated in de Cappet's argument—but the probability of one forming spontaneously is small but definite, and it is not necessary to add one.

Q. When you say that the probability is small but definite, I assume that we are now drifting over into the question of whether there is anything absolute or not. I take it that there is a small but definite probability—a definite and mathematically ascertainable probability, that we will have a fall of meteorites here this afternoon. A. I think that there are several reasons that I can use to support my position. The bottom of that same page that you referred to, "the supersaturation curve of the Miers type are difficult to reproduce except under carefully controlled conditions."

Mr. Lutz: Dr. Lowry, will you please refer to the line you are reading from?

The Witness: The last line on the first column of page 1055, and continuing for the first three lines on the top of the second column. Shall I repeat what I read, or—

Q. No, sir, I would like you to read the next three lines after the passage you picked out. I assume, of (819) course, that you are going to disagree with those three lines. A. (Reading): "The supersaturations obtained in

seeded solutions are definitely considerably less than those found in crystallizing unseeded solutions."

That is exactly one of the points that I wish to make, that the supersaturation that can be obtained is dependent upon so many experimental conditions that they are not reproducible, and as an illustration of that fact, I do not think you can find in any handbook the supersolubility of any substance; while you can find the solubility of the substance, you cannot find the supersolubility. It depends upon the experimental conditions.

Q. Dr. Lowry, do you agree with that statement in those three lines or not? A. I certainly do.

* * * * *

(823) Q. Doctor Lowry, in your understanding of the term seeded solution, can such a solution exist without a crystal being present? A. I would say no.

* * * * *

(825) That's all right. Now just answer this question. I see nothing unclear about it. Can there be such a thing as an unseeded solution which contains a crystal? A. If you accept my definition, no

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Deposition of Carl Otto.

CARL OTTO, the plaintiff, examined before trial by defendants, being duly sworn, testified as follows on direct examination by Mr. Brown: This testimony was offered in evidence by the defendants.

* * * * *

(O. 6)* Q. What, if any, is your connection with the German Otto Company at the present time? A. I am practically it; I am the main shareholder of the German Otto Company.

Q. Do you operate in the building of by-product coke oven (O. 7) apparatus through a corporation? A. Do you mean through other corporations?

Q. Through a corporation in the United States—through any United States corporation. A. I have this Otto Construction Corporation.

Q. What is your relation to that Otto Construction Corporation? A. Of the Otto Construction Corporation, I am now two-thirds shareholder, and my son is one-third shareholder.

Q. Are you the president of that company? A. I am the president of that company.

Q. Is that company a licensee under your patents? A. Yes, that's right.

Q. Is it an exclusive licensee? A. No, we didn't make a special agreement with that. You see, I think that was never made. That is because I am the owner, and that is the owner corporation, but it is the understanding that they have the full license to use it.

Q. That is just an oral permission, if I understand correctly? A. That's right.

Q. Without any formal license? A. There is no formal agreement made.

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* References to pages taken from deposition testimony are preceded by letters:

"O"—Deposition of plaintiff, Dr. Otto, taken at New York, N. Y., December 20 and 21, 1954;

"B"—Depositions of Beatty and Toohey of Bethlehem Steel Co., taken at Bethlehem, Pa., February 14 and 15, 1955;

"W"—Depositions of Byrns and Phillips of Woodward Iron Co., taken at Birmingham, Ala., November 22, 1955.

Q. In other words, the corporation, in effect, is you, operating to build apparatus? A. That's right; it is practically a unity of persons.

(O. 8) Q. Have you granted any licenses under the patent in suit to any others than to Otto Construction Corporation, about which you have spoken? A. No. Do you mean this patent which is now in the suit here?

Q. Yes. A. No.

Q. Have you been asked for a license by any company in the United States under the patent in suit? A. Not yet.

Q. Have you sued any company in the United States under the patent in suit, other than the Koppers Company and the Wheeling Steel Corporation? A. Nobody else.

Q. Have you given notice of infringement to any other company than the two defendants—of infringement of your patent in suit? A. No.

Q. So far as you know, as you are advised, are there any other companies infringing your patents in the United States by building apparatus that you think comes under the patent in suit? A. Yes; that's right.

* * * * *

Q. You have taken out a large number of United States patents? A. Yes, that's right.

Q. Do they all relate to by-product coke oven structure? A. To coke ovens and by-product structure.

(O. 9) Q. I think I saw a statement in some paper that I read, that, prior to the filing of application which resulted in the patent in suit, you had filed five other applications relating to spray-type saturators for removing ammonia from coke oven gas—(O. 10) is that right? A. That's right. You see, the first patents I made myself—the first patent application—and then, later, I convinced myself it is better I take an expert who knows how to draw up the patents.

These are mentioned, I think, in this patent which I mentioned. This refers to the other applications.

Q. Well, is it fair to say, then, that, over a period of time, you were working to develop a spray-type saturator? A. That's right.

Q. And that these prior applications represented other forms, or whatever you want to call them—steps—in the development which led up to the patent in suit—is that right? A. That's right. That is a continuancy in it, because I understand that this patent in suit also refers to this.

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(O. 12) Q. In the file history, there are some affidavits by certain officials of the Bethlehem Steel Company, if I recall—two of them, if I recall rightly. A. That's right.

Q. Can you tell me who prepared those affidavits—who wrote them up before they were signed?

The Witness: (Addressing Mr. Hubbell) Did you prepare them?

A. I don't know if Mr. Hubbell prepared them.

Q. Is your answer that you don't know? A. I won't say that. I think Mr. Hubbell worked with this, too. I know that. I don't remember quite that performance.

Q. Do you know who collected the information that is referred to in those two affidavits in your file history? A. What is that?

Q. Do you know who collected the information? A. The information, for example, you see, that we, of course, put up, was to show the importance—to show there were, before, so many saturators, and later on, so many saturators. That was the idea that came from us—to show the importance of the invention.

Q. Well, now, that is the object. You have stated the object of the affidavits. What I want to know is, who pre-

pared those affidavits? That is the first question. (O. 13) A. I talked with Mr. Hubbell about that.

Q. Did you prepare them, or did Mr. Hubbell prepare them? A. I didn't prepare them. Mr. Hubbell, I think, did.

Q. As far as you know, Mr. Hubbell did? A. Yes.

Q. Now, do you know who interviewed the men that made affidavits, preliminary to preparing the affidavits? Was that you or Mr. Hubbell, or somebody else? A. I think I approached these men.

Q. And then reported to Mr. Hubbell? A. And I reported to Mr. Hubbell. I don't think that Mr. Hubbell ever saw them.

Q. And, I assume, that was shortly before the affidavits were executed? A. Yes, that is so.

Q. But Mr. Hubbell wrote them up in formal language, I assume? A. That's right. I think you will see that from the wording.

Q. When did you last have any contact yourself, or through anybody else, with Mr. Robert Pugh and/or Mr. Harold Cox, whose affidavits were introduced in the file history? Do you understand the question? I know it is a little involved, and I will have it read back, if you want it. A. Do you mean, when I last saw or had contact with these gentlemen?

Q. Yes. (O. 14) A. Do you mean now, or before they made the affidavits?

Q. As of now. A. You see, they were superintendents. Let me explain. Keep separate Mr. Cox and Mr. Pugh. I talk now about Mr. Cox, because we have to keep that separate.

Mr. Brown: I'm sorry. I didn't mean to mislead you. I should have kept them separate.

A. (Continuing) We cannot generalize. Mr. Cox is still the superintendent of the Johnstown plant, and I saw him in other matters last, not with this affidavit.

Q. When was it that you saw him last? A. Oh, at a meeting, I think that was—a coke oven and blast furnace meeting. I think that was a meeting in—I think that meeting was in Johnstown.

Q. When was it? A. I saw him in different matters here. Also, I saw him when I made a visit to Johnstown, and I talked to him, for example, about coke oven doors—something quite different; nothing to do with this story.

Q. Then, is it fair to say that you had seen Mr. Cox quite frequently in the last two years? A. I wouldn't call that frequently, you see, when you see him, perhaps, two or three times in a year.

Q. Every year? A. Two times, perhaps, especially at the meetings.

(O. 15) Q. Did you, at any of those meetings with Mr. Cox, discuss the suit brought by you against Koppers and Wheeling? A. No.

Q. Did you ever discuss his affidavit with him after he executed it? A. I just expressed to him my thanks that he gave us the affidavit. That's all I did, which is quite natural.

Q. Now, as to the other man, what contact have you had with him since he executed his affidavit? A. Mr. Pugh? You see, with him I had very close contact because he left the Bethlehem Steel Company, and we engaged him as an engineer, and when he was in our office, and there, in his work, of course, I had then contact with him about the work that was to be done in our office. Then, of course, you have close contact, you see.

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(O. 16) Q. Did you ever ask Mr. Cox to come and work for your company. A. No. We didn't ask Mr. Pugh, because

Mr. Pugh had to leave the Bethlehem Steel Company, and we were looking for engineers, and then we approached him. We didn't put any incentive there to leave the Bethlehem Steel Company.

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(O. 17) Q. Is my memory correct that you said that Mr. Hubbell had never seen Mr. Cox and Mr. Pugh, so far as you knew? A. Yes I think he never saw them.

Q. Did you know a Mr. Underwood of the Bethlehem Steel Company? A. Oh, yes, sure, I knew him, because he gave us a first saturator order for a cracker pipe. That was another construction.

(O. 18) Q. Did Mr. Pugh and Mr. Cox work under Mr. Underwood for the Bethlehem Steel Company? A. No, Mr. Underwood was the superintendent at Bethlehem—at the Bethlehem plant of Bethlehem Steel—and he was the superintendent of the coke ovens in Bethlehem, while Cox was the superintendent of the coke ovens at Johnstown, and Pugh was the superintendent of the coke ovens in Lackawanna.

Q. Was Mr. Underwood, so far as you know, in any way a higher official in an engineering capacity for Bethlehem than Mr. Cox and Mr. Pugh? A. No, I don't think so, because I understand the situation so that they all were superintendents of different plants; that they have a coke committee, and I understand that at that time Mr. Underwood was the chairman of this committee.

Q. Dr. Otto, do you expect to be a witness for plaintiff at the trial of this case? A. Do I expect that? I don't know how that goes on. I think that is with the Court and, of course, if they ask me, I certainly will be a witness because I want to do everything in favor of our interests.

Q. Mr. Underwood was chairman of the coke committee of the Bethlehem Steel Company, at the home office, wasn't he, to your knowledge? A. Yes, that is what I just told

you. That is what I understood, but I understand that they always select, sometimes (O. 19) this gentleman, and sometimes another gentleman.

Q. Well, I am asking just for your information. Isn't it a fact that Mr. Underwood had a very large part in determining what coke oven apparatus would be installed by Bethlehem in all of its plants, or any of its plants? A. Well, because he was—has been—very long with the Bethlehem Company, and once, I tell you this, we found out, to our own damage, when this order for the coke oven and steel went to Koppers, that it was due to him.

Q. I think the answer to my question is yes, isn't it? A. Well, I think he had some influence there. They listened, in any case, to what he said.

Q. Did Mr. Underwood ever turn over to you data regarding his own experiments in connection with the saturators for removing ammonia from coke oven gas? A. No, not data. I remember, he talked about what happened there with the plant after we built it. Not only he, but, I think, all the people at Bethlehem were very cooperative. They didn't keep secret what was going on with the saturator.

Q. Did Mr. Underwood ever turn over to you, notes and data about his own study of saturators for removing ammonia from coke oven gas? A. No, I wouldn't say that.

Q. Do you know whether or not he had worked on the subject matter of removal of ammonia from coke oven gas for a good (O. 20) many years before you put in any installations for Bethlehem? A. No. I remember there was a patent once on Underwood, but I wouldn't say it was a saturator—was it a saturator, or something that he worked on—but I don't remember that I talked with him about it.

Q. Your answer, then, is that Mr. Underwood never discussed with you before you made an installation for Bethlehem of a saturator for removing ammonia from coke

oven gas, that general subject matter? A. No, no. I didn't talk with him especially.

Q. How long have you known Mr. Underwood? A. I saw Mr. Underwood when I came over, you see. Of course, I visited all the different plants. I tried to visit not only Mr. Underwood but other plants. Then, I saw him. That must be, perhaps, I saw him already in '39, '40. And then I tried to keep contact with him, but I only remember that he reacted very sour.

Q. Did you see him rather frequently from 1939 on? A. I saw him then frequently after we have built the plant, because I went over there to the plant, and it was the general practice, before I went to the plant, to say "Hello" to the superintendent.

Q. Didn't you see him rather frequently before you put in any apparatus for Bethelehem of any kind? A. No.

(O. 21) Q. Dr. Otto, in the development of the apparatus which is described in the patent in suit, on which you have said you had at least five prior applications filed— A. (Interposing) That's right.

Q. —did you work with any engineer or other person in that development? A. I made most myself. You see, I talked, perhaps, sometimes with my son, but I didn't talk with other engineers.

Q. Then, the development— A. (Interposing) Was entirely made by myself.

Q. And you didn't consult anybody, at all? A. No.

Q. In that development? A. No.

Q. Didn't you go around and talk to anybody? A. After I had developed it, you see, then I tried, of course, to sell it—to try to get it in practice.

Q. Now, when do you regard your development, as embodied in the patent in suit, as having been completed by you? A. I didn't quite get that.

(The last question was read.)

A. Do you mean the time?

Q. Yes. A. In general, I tell you this: I would say about April, May—in '46 or '47—when was this first patent going in?

(O. 22) (Off the record.)

A. (Continuing) I guess, about April or May, 1946, and then I also remember when I made the first effort to sell it, because we had then in our office Professor Simmons and Professor Stahl, from the Research Department of Bethlehem. To them I even showed my patent drawing before the patent was filed. I said, "I show you this only confidentially."

Q. Is that the patent drawing of the patent in suit that you are talking about? A. That's right. You see, I had the patent drawing—it was either this (indicating)—

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Q. What drawing is that? A. Of my application.

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A. (Continuing) You see, application was made on June 4, 1946. But, as far as I remember, I showed that drawing to the Bethlehem people earlier.

Q. I ask you, again, was that one of the drawings that is now in your patent in suit? A. That patent is not in the patent in suit, because—wait a minute. (O. 23) Yes, maybe not quite accurately, because this drawing I made myself.

Mr. Morton: In referring to this drawing, the witness refers to a drawing labeled "Figure B," in his file of the proceedings in Application Serial No. 67344.

Mr. Brown: Is that one of the abandoned applications?

Mr. Morton: It is one of the abandoned applications.

Q. And you didn't show any of the drawings that are now in your patent in suit—is that correct? A. That is correct, because they were made later. But they were very similar to it. As a matter of fact, I think they are practically identical, if you look at these two drawings.

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(O. 26) Q. Dr. Otto, have you kept yourself familiar with the literature relating to by-product coke ovens, and particularly with relation to the removal of ammonia gas from by-product coke ovens? A. Yes, I did.

Q. Has that been true for a great many years? A. Oh, yes; always. You see, it comes in the business automatically, just being there.

Q. Were you familiar with the literature and the practical art in Germany even before you came to the United States? A. That's right; oh, yes.

* * * * *

(O. 28) Q. Now, do you know of any other situation, aside from removal of ammonia from coke oven gas, where apparatus and methods are used for removing one gas from another gas? A. You remove, also, benzol from the coke oven gas. You see, you remove naphthalene, and you remove tar. You see, in the other fields, these are many possibilities where you remove, also, in the oil industry, and, for example, I don't like to forget that you remove H_2S. You remove that. And cyanates, you remove. You see, that is a very big field, if you are going into that one.

Q. Before you completed the invention which you allege is covered by the patent in suit, did you know of the use of sprays for scrubbing gases and separating gases? A. Do you mean, the use of sprays for scrubbing?

Q. I am using the terms loosely, and you can define them to suit yourself. I think you will know what I mean. A. The use of sprays, I certainly knew that—the use of sprays. And especially I knew them in connection with towers which are filled with grids.

(O. 29) Q. Did you know, from either practical experience or your knowledge of the literature, and that includes patents, of the use of sprays for separating one gas from another, or for taking some element out of the gas? I am talking about sprays, now, particularly. A. Well, sprays, for example—you know the Felt-Warner. There you use—it was a very successful scrubber. You see, they take the liquor which flowed in from the top and made it distribute by a rotating movement, and took, then, for example, the benzol out; also, they could take, with water, the ammonia out. You see, that was a very successful scrubber.

Q. And those scrubbers that you are talking about, you knew about? A. That they didn't use sprays. They divide up the liquor by rotating means.

Q. Did you know of any use of sprays for removal of one element from a gas, prior to the completion of the invention which you say is covered by the patent in suit? A. I knew of the grid brushers, you see—the grid scrubbers, you would say. You see, where you have to distribute the sprays in the top.

Q. Is that the only use you knew of sprays before you made what you— A. (Interposing) That's right. The Otto Company had a big (O. 30) spray, that, is, also—you find that in the patent application. That is called a splash spray. That is a spray which is accepted in—I think Wettly uses it in the patent on that. That is a splash spray, like an umbrella, and to distribute the liquor over the grids. For example, the Otto Company, in Germany, they had a tower, a scrubber, where they had sprays in different

sections, to distribute the gas. And then they had grids below it.

Q. Is that all you knew, from your reading and experience, prior to the date you have given as completion of the invention of the patent in suit? I am referring to the use of sprays. A. That's right.

Q. Is it your understanding that your patent discloses anything not previously known to you and in the art, to your knowledge, as to the production and use of sprays? A. Sure.

Q. Well, I don't understand your answer. Sure, what? A. I didn't know that that was done before. I do not know that sprays, in this way, as I did it, were ever used before. I also never had before, this idea, because we had to do something here to improve our construction. You see, in Europe, much more went on with the old cracker pipe saturator.

Q. I don't want to interrupt you, but, if you have answered all you want to answer, I will ask you another question. A. Is that enough answer for that?

(O. 31) Q. That is for you to say. You answer all you want to. A. Yes. You see, I didn't know anything before.

Q. Well, now, you knew that you could go out in the commercial market, long prior to 1946, and buy spray nozzles that would give you practically any kind of spray you wanted, didn't you? A. Yes, but I didn't know that I could use a spray, especially for the mother liquor. I was myself very hesitant.

Q. Well, isn't it a fact that the sprays you used in the first spray-type saturators you made, were sprays that were on the market and could be bought by anybody? A. Sure, sure. I knew that there were sprinkler sprays.

Q. And didn't you know that there was a company that would give you a spray nozzle that would give you practically any kind of spray you wanted? A. Yes, because,

especially I can tell you, I was very pleased. I can tell you, I developed the idea even in Germany, many years before, for a spray with a whirling action, with waves in it—in Germany—and we tried it there. I was very pleased that in America you could buy these sprays just on the open market without any difficulty, and I happened to think about, if the spray would work or not work. I knew the spray would work.

* * * * *

(O. 32) Q. When you started to use the spray, all you had to do was go out in the open market, and you could get a spray that would give you any kind of spray as to fineness, or diffuseness, of the emulsification—whatever you want to call it—of the liquid? A. That's right.

Q. And the mere fact of the use of spray nozzles for giving you any desired degree of fineness of spray, was well known in the art as something that you could get on the open market? A. That's right; absolutely.

Q. And didn't you know that these sprays were used for producing any desired fineness of spray, for projecting that spray— A. (Interposing) I knew that.

Q. —for projecting that spray into an open chamber. A. I knew that. The only thing I did not know was the use of the spray for mother liquor, where slurry comes in, and liquor comes in, and slurry can be very thick. I was very glad to find out that these sprays never clog up. That, I did not know.

Q. You didn't know it, but when you went out and bought one, you were able to get a spray that would do exactly what you wanted it to do, by purchase in the open market? A. That's right.

Q. Well, now, reverting a little, what were these sprays used for, prior to your alleged invention—I mean, spray (O. 33) nozzles? A. Especially for cooling.

Q. Gases? .A. For cooling air or for cooling, also, water. You know, in the spray towers, they are used a lot, commonly.

Q. Didn't you also know that they were also used in closed tanks or compartments for spraying a liquid through a gas, for treating the gas, prior to your completion of what you call your invention? A. I am telling you that they were used for distribution of the liquor in scrubbers which were not empty—that means, in the coke oven field I talk about now. You see, where they always—there were grids in, to give a bigger surface for distributing.

Q. I am not sure you answered my question, so I will ask it again: did you know, through literature or otherwise, of the use of sprays for discharging a liquid through a gas, for any purpose? A. For any purpose? Sure. That I knew, that a spray—just you spray into a gas, especially in air—they are mostly used for water, for cooling water. That is well known.

Q. Well, you would, in a general sense, regard air as a gas, wouldn't you? A. Oh, yes.

Q. Then, you did know of the use of sprays for discharging (O. 34) a liquid, in finely divided particles or globules, through a gas, before you started to use sprays in connection with coke oven gas—is that right? A. That's right.

Q. Well, isn't it fair to say, Doctor, that the use of sprays for discharging finely divided liquid through a gas, was a tool of the art, well known to chemists and metallurgists, and physicists, long before any date that you claim for your invention in this case? A. That's right.

Q. Don't you also know that in the literature the use of sprays for discharging liquid through coke oven gas, was shown and known? A. Sprays are known.

Q. You say that it is known. I am talking about before you claimed to have made your invention. A. That is known, but I also knew that in the coke oven field, always grids, also insets, were used, to get the contact surfaces. That, I also knew.

Q. Well, now, as I understand the first part of your answer, you admitted that the use of sprays to project a liquid in finely divided form through coke oven gas, was also known prior to any date you claim for the invention of the patent in suit—is that right? A. Yes.

(O. 35) Q. Well, now, the purpose of a spray, generically, is to give you a finely divided state of the liquid, for the purpose of giving you a desired surface contact of the liquid with the gas, isn't it? A. Yes, that's right. Yes, sir, I did.

Q. That was well known to physicists and chemists long before any date that you claim? A. Yes, but they never tried to do it exclusively with sprays. They only used the sprays to divide it, and then had other means to make the contact.

Q. Well, your answer to my question is yes, and then you add this other—is that right? A. That's right. And I add this other because this is important.

Q. Now, you also knew that the use of sprays for dividing a liquor, or liquid, to get optimum contact of liquid and gas, was also known in the coke oven gas field, did you not? A. Do you mean, exclusive only with sprays? That was not known.

Q. I didn't say exclusive. I am just talking about the use of sprays. A. For dividing, for splitting up, but not for taking out exclusively only these particles from the coke oven gas.

Q. But you knew that sprays were used for discharging liquid in finely divided form through coke oven gas, prior to your (O. 36) alleged invention? A. That's right. By this

they got the distribution for other means. That was the purpose.

Q. Of course, you understand, Doctor, we talk in broad terms, and I am talking about the use of sprays to give you a large degree of contact between liquid and gas, and you have said that that was known also in the treatment of coke oven gas—is that right? A. That you can get a bigger contact, but nobody did it.

Q. Well, I am not sure you have answered my question. A. I don't quite see what you are driving at, because I understand you think that it was known in the coke oven practices, also, in scrubbers—to use it, and I use now, again, the word "exclusively," to get this contact.

Q. That isn't what I asked. A. That was never asked.

Q. I am asking you a very broad question, now, and I will tell you what it is for. Maybe I will state it, and then you correct it. My understanding is, that in the chemical field, in the gas treatment field, or physical chemical field—whatever you want to call it—the spraying of a liquid through a gas, to get finely divided liquid, to get a desired amount of contact of liquid and gas, was known in the art long before your alleged invention. Can you say yes or no to that? A. Because there we come already in a field what we know (O. 37) now, you see.

Q. No. I am talking about— A. (Interposing) You talk about the past.

Q. I am talking about the past, before you made your invention. A. Yes, and that was not known to me.

Q. When you spray a liquid through a gas, where the liquid is such that it absorbs something from the gas, you knew, before your alleged invention, of the use of such sprays for that purpose, didn't you? A. That's right. But it only absorbs in that part where they extend, and then the drops fall down, and have to fall on other means, like grids, where then the work has to be done. That is the

coke oven process or practice, or was the coke oven practice.

Q. You keep answering what seems to me to be a complete answer, and then saying something else. Did you know, in the art, or otherwise, before your alleged invention, of the use of sprays for treating coke oven gas, aside from the particular use of grids, and so forth, that you tacked on to your last answer? A. Yes, to do alone the work, I didn't know, and even to date people question it very much that you can do that, with sprays alone.

Q. Doctor, if somebody had asked you in 1945, which, I (0.38) understand, is before any date you claim in this case, what would be the effect of spraying a finely divided liquor comprising sulfuric acid, through coke oven gas, would you have been able to tell them what you would expect the results to be? A. I didn't say that I expected it would really do the whole job, but it really did. I did not know the efficiency.

Q. All right. You didn't know because you hadn't tried it? A. That's right.

Q. But, if somebody had asked you, say, in 1945, or 1940, whether or not spraying of these liquors that we are talking about, through coke oven gas, wouldn't you have known and been able to say that it would absorb a large part of the ammonia? A. It would absorb, but I would say there would be big troubles, because this liquor, you see, in this saturator—this clogging up, and also the encrustments, is a big difficulty which has to be overcome, and I would be very hesitant—and, also, when we put up the first saturator, you see, I was very pleased that it worked as it did.

Q. Now, how did you solve this difficulty that you are talking about, in the apparatus described in the patent in suit—this difficulty of clogging up? What did you do to solve that? A. You see, I provided that it could be

flushed. I saw that about the walls, and, even, I tried to keep it as far empty as possible, and by this we really succeeded to have—to build—(O. 39) a saturator which practically had never to be killed. To be "killed" means cleaning. We put special attention to it, because we knew from our practice—we knew in the cracker pipe saturator—this rock salt formation is very unpleasant, and you can have very sleepless nights, and you stand in front of this thing and the gas doesn't go through, and everything is blocked up.

Q. You say that one of the things you did was to wash the walls—was that it? A. That's right. You see—

Q. Was there anything new about that? A. It also was shown in there, in my patent application. I mentioned things like that.

Q. Didn't you know, prior to— A. (Interposing) It was killing; that I knew.

Q. Didn't you know before your alleged date of invention, that washing had been used to remove or prevent salting on the walls of apparatus for removing ammonia from coke oven gas? A. Walls to be washed? That we found out ourselves in our first cracker pipes.

Q. What you knew, and not what you found out. A. No. I only knew about the diluting of the bath. That was the normal procedure.

Q. Well, then, do you think that the washing of the walls is an important feature of your invention? (O. 40) A. That is one.

Q. And new with you? A. That was practically new with me.

Q. What do you mean by "practically"? A. Well, you see, that, I had. I kept at the wall a certain velocity, you see, and we had a certain velocity of the liquor running over the wall. The salt cannot stick so easy as if the liquor is stagnant.

Q. What I don't understand is your statement, which, as I remember it, was that it was practically new. What do you mean by "practically new"? A. I never knew that this feature had been used before—just to make liquor run down the wall, with a certain velocity, and then have the other drops, for example, fall into that liquor.

Q. Well, without qualifying as you have, didn't you know that washing had been used in connection with coke oven gas ammonium removing apparatus? A. Washing, that's right; with water, that's right.

Q. It was known before your invention? A. That's right; to wash it out.

Q. Now, aside from washing, what did you do to avoid the difficulties which you say had confronted you in using this spray-type of getting contact of the liquor—we will call it—with the coke oven gas? I think you said in the beginning that (O. 41) you knew sprays were old. You knew sprays were old even for coke oven gas. But you were confronted with certain difficulties. A. That's right.

Q. I asked you how you knew of these difficulties, and as far as I recall, the only one you referred to was washing the walls. What other difficulties did you meet, and how did you solve them? A. Difficulties, to have a spray which was nonclogging, and I picked the spray which was known in the art, you see, this special Eneas construction, and where I expected to have no difficulty with clogging up. They call it a nonclogging spray.

Q. As I understand it, you went out and picked out, in the art, a commercial spray which would give you what you wanted—a spray nozzle? A. One which I hoped to give me what I hoped to get. You see, it could just as well happen that this spray, for example, would have, maybe, clogged up.

Q. Did it give you what you hoped to get? A. Yes, it gave me, because I don't remember that that spray—that not a single spray in our saturator has ever blocked up.

Q. And the spray that you bought on the open market gave you the results you wanted, without any modification by you—is that right? A. That's right.

(O. 42) Q. Your patent speaks several times about spraying liquor through an unobstructed space. Just what do you understand or what do you mean to say by those words? A. That means, empty. As I understand it, it is empty space. That means, no grids, or other filling, you understand, or wooden grid.

Q. Well— A. (Interposing) That is well known in the art, where you can put in, for instance, tiles.

Q. Well, then, if you sprayed liquor into any tank which was an open tank—by that I mean, just, say, cylindrical, for example—and then the liquor collected at the bottom, that, to you, would be spraying it into an unobstructed space, would it not? A. You just spray it in. If you don't spray it to have it distributed above, you need some other means to make the contact.

Q. Well, does that mean that there is nothing to bar, or interfere with, the spray, between the time it leaves the nozzle and the time that it reaches a liquid surface below? A. That it reaches a liquid surface and has done all the work before; not that this spray then reaches a surface, say? for example, grids, and then really to do the main work. It is difficult, you see, to explain that from my side—how I look at that.

(O. 43) Q. I think, Dr. Otto, that you are taking my question to mean more than I did, and making it more complex than necessary. I want to know if what you meant by an unobstructed space was simply that your spray went into a gas chamber so that the spray could pass through the chamber without having to go to other surfaces, other than the walls of the tank, until it reached the liquor at the bottom of the tank. Is that what you mean? A. That's right.

Q. Now, do you know, in the art, of any case in which sprays are shown and illustrated, where the spray is injected into a tank which has cylindrical walls, for example, and the spray is unobstructed and unimpeded in any way until it reaches the bottom of the tank? A. Until it reaches the bottom of the tank? Just spray—you have to go back now to the Jeremiassen patent. You have jet sprays.

Q. No, without reference to any patents. I am asking you just the general question. I will put it this way: don't you know that the art shows sprays which projected a liquor into a tank or compartment—whatever you want to call it—in which there were no baffles or grids, or anything else? A. Just a spray.

Q. Until the liquid came to rest at the bottom of the receptacle—whatever you want to call it. (O. 44) A. Those sprays are used in pipelines, sure.

Q. Is your answer yes? A. Yes, that is known.

Q. Well, then, as I understand it, the words "unobstructed space," in your patent, mean simply that the sprayed liquor could pass directly from the spray nozzle down through the compartment to the point where the liquor is first collected—is that right? A. That's right.

Q. And if there was any obstruction in there, such as a baffle or a partition across the tank, then that would be an obstruction—is that what you mean? A. That's right.

Q. Doctor, didn't you know before your completion of the subject matter which is described and explained in your patent in suit, that the absorptive effect of liquor passing downward through an unobstructed receptacle through which coke oven gas was passing, would depend in absorptive effect somewhat on the fineness or coarseness of the spray? Now, if you don't understand, we will have the question read back.

(Off the record.)

A. I want to say, somewhat; that is somewhat part of the efficiency. I think that is known.

Q. You know that a fine spray— A. (Interposing) A fine spray would absorb better than a (O. 45) coarse spray.

Q. But, if there was enough coarse spray passing through the gas, it would absorb substantially all of the ammonia from coke oven gas, would it not? A. You mean, coarse spray?

Q. Yes. A. It would not have that efficiency of a fine spray. That is logical.

Q. That is, you could use less of the fine spray, but, if you used enough coarse spray, you could take substantially all of the ammonia out of the coke oven gas? A. If it was big enough, possibly, yes.

Q. Then, isn't it fair to say that it is a matter of judgment and a matter of degree as to how fine a spray you used or how coarse a spray you used? A. Well, you see, there are also other points. Then you come to segregation—that the gas carries the fine spray with it. There are other points which come up, which you have to consider, when you go into this matter; the velocity of the gas.

Q. In the apparatus that you build at the present time, do you use both a coarse spray and a fine spray? A. You see, we have both in there. One spray is smaller. The smaller sprays are finer; and we have very big sprays.

Q. And that is true of all of the saturators you have built (O. 46) for removing ammonia from coke oven gas? A. Yes, for both types; it would work with both.

Q. Do you regard the use of both fine and coarse sprays as essential, to make your apparatus operative? A. No, no. That is not just—you can't—for example, if you would only put in fine sprays, then your carry-over is much more, and you put more load, for example, on the acid catcher.

Q. Doesn't that come back to my original question, that you could use either coarse sprays or fine sprays, if you

wanted to? A. Yes. It is not limited to coarse or fine sprays.

Q. And it is a matter of what you want to do and the degree of what you want to do? A. Yes. And the conditions, also.

Q. It is an engineering problem as to how much gas you are treating and how much liquor you are using? A. Yes.

Q. And you could use all coarse? A. We could use all coarse. Then you have to put more.

Q. Or, conversely, you could use all fine spray? A. That's right.

Q. But you would get somewhat different results, if I understand rightly? A. That's right.

(O. 47) Q. The real thing you are after in your spray through a coke oven gas, is to get enough contact of liquid and gas, is it not—to make the necessary contact for the special purpose? A. That's right.

Q. And the working out of the amount of spray, the coarseness of spray, the flow of gas through the tank where it is exposed to the spray, is an engineering problem that has to be worked out? A. That's right; it has to be worked out.

Q. In each particular case? A. That's right; there are all these problems for each case.

Q. Well, do you think that your patent discloses anything that is critical and essential as to the fineness or coarseness of spray? A. I don't think so; not critical. It would be, for special purposes, critical. I mean, for example, you know, in my patent. But that is just one claim—with the top spray to make it correspond, to have less carry-over, to hit some mist out, you see.

Q. Well, then, Doctor, if I understand you correctly, for any particular installation, do you have to tailor it, as a matter of engineering? A. That's right, that's right, as a matter of engineering.

Q. Then, you have to tailor it according to local conditions? A. I can tell you here—I can tell you that, for example, in (O. 48) one place we have replaced the spray nozzles, and the top, for other types of nozzles, to have a special coarse spray. I can tell you that, for example, to hit out easier the smaller particles.

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(O. 51) Q. Doctor, when you say that you decided to use, or intended to try out, the spray type of saturator, involving the spray nozzles, and that you were confronted with certain difficulties (O. 52) which you had to meet, and I think you have named two—one was washing the walls, and the other was nonclogging spray nozzles—now, what other problems confronted you when you— A. (Interposing) Well, of course, washing the walls—

Mr. Brown: I think it will help you if I finish the question.

(The last question was read.)

Q. Continuing the last question: when you decided to build a saturator for removing ammonia from coke oven gas by spraying liquor through the gas, what other problems— A. (Interposing) Well, for example, you get better efficiency. That was, of course, the main problem. You see, of course, you said, already, you can make these nozzles very fine, and, of course, now, that is very obvious. Now, it does, efficiently. But I tell you, when I first saw that drawing in Lackawanna, the cracker pipe saturator was described like this, because Bethlehem insisted that the saturator, in case it won't give the efficiency, would be able to be changed in a cracker pipe saturator. The efficiency was the very main point.

Q. Well, now, the determination of the efficiency of the use of sprays through an unobstructed space was a matter

of cut and try, and experiment, was it not? A. That's right. You see, it looks very easy now.

Q. Have you made any alterations in the apparatus which you (O. 53) first built, embodying, as I understand it, the subject matter of the patent in suit—since the first? A. No. You see, the only change we made in Lackawanna was that we added one spray. We did not have a middle spray. That showed the efficieney was not quite perfect, and we added an additional spray in the middle, and then it was all right.

Q. Now, that, I assume, was because you were not getting enough spray for the volume of gas? A. That's right. It was not quite enough.

Q. What you did was to add another nozzle, and produced more spray? A. But the possibility for this was provided for, you see. We had figured that coming possibility.

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Q. I believe your patent, by the drawings, shows five different arrangements of apparatus. Have you built all of those (O. 54) arrangements that are shown in the five figures of your patent? A. No, not all.

Q. Which of them have you built? A. You see, we have built this.

Q. Give the number of the drawing, please. A. Figure 1, of my patent.

Q. You have not built that? A. I have built that.

Q. Oh, you have? A. That was built in Lackawanna.

Q. Figure 1, at Lackawanna? A. Yes, at Lackawanna. Figure 2 was not built.

Q. Was it ever built? A. Never. Well, then, Figures 3 and 4, with adjustments, have been built in Johnstown and Ford, and Morgantown. This saturator—

Q. Which saturator? A. —No. 5, was not built, and I tell you this: it wasn't built because it would infringe the

Jeremiassen patent. Not the Jeremiassen patent which you have in mind. I will tell you the feature: it would infringe to have a central pipe going into the crystallizer.

Q. You are referring to a Jeremiassen, United States patent? A. Yes. This is another patent.

Q. I noticed that you passed over No. 4. What is that? (O. 55) A. Nos. 3 and 4 are more or less the same, and with some modification these were built in Johnstown and Ford, and Morgantown, and Indianpolis.

Q. Which of these four forms are arrangements that are shown in your patent drawing? A. Nos. 3 and 4 are practically the same.

Q. That is the reason I am saying "four" instead of "five." Which of these four forms of apparatus that are shown in your patent drawing do you regard as the best and most effective for your purposes in carrying out the process of the patent? A. Well, I think we make always improvement at this No. 3 and 4, because nobody would build a scrubber like we built it, before—the first one. A. scrubber is mostly built as a cylinder, parallel, but we did it the other form, because Bethlehem wanted us to build it in such a way that we could go over to a cracker pipe.

Q. Which one of them did you pick as the best? A. The 3 and 4.

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(O. 56) Q. Now, Doctor, was there anything new in your patent, in your opinion, in the tar catcher? A. What is that?

Q. The trap for catching tar and eliminating it. A. You mean, the tar from the overflow?

Q. Tar; yes. A. Oh, yes, there is nothing special.

Q. That was an old and conventional unit? A. Yes, the tar just came off.

Q. Do you use the heating jackets in your apparatus,

as they (O. 57) are shown in the figures? A. Not as they are shown, because that is too expensive. We use, now, copper pipes. We have copper pipes around and we put the heat in there.

Q. But, you do use heat with all of them? A. That's right.

Q. Is it essential for the practicing of your patent? A. It makes it easier. It is difficult to run without it.

Q. Well, now, Doctor, I notice in these several drawings of your patent that you have what I will roughly call crystallizer receptacles, placed at a number of different positions with reference to the spray chamber—is that right? A. That's right.

Q. Does it make any difference where your crystallizing chamber is located with reference to the spray chamber? A. It doesn't make much difference.

Q. It can be below or to the side of, or even at the top, if you pumped it up? A. Oh, sure, if you pumped it up, you could. It is only a question as to the pump that you would have, and that Koppers should know, because they have done it.

Mr. Brown: Perhaps they do know it.

Q. And that is true, also, of the acid catcher and the tar trap? A. Wait a minute. Do you mean the acid catcher?

(O. 58) Q. Yes. A. That is conventional, isn't it? This cyclone acids catcher is the same as with the cracker pipe.

Q. And that can be at the top or the side, or any convenient place? A. Oh, yes, because, as a catcher, I think they are more or less all the same.

Q. In fact, they are conventional with any kind of apparatus for treating coke oven gas? A. Yes; there are many constructions.

Q. The same is true about the tar separator? A. Yes; you could say that. You see, there is nothing I would say is revolutionary or special there.

Q. As I understand, also, from the variety of forms you have shown, you can position the crystallizing chamber at any convenient point? A. That's right.

Q. And you can use one chamber or two chambers, or a multiple of chambers? A. That's right.

Q. And, so far as your interpretation of your invention is concerned, it doesn't make any particular difference where you place your crystallizing chamber? A. No, it doesn't make any difference.

Q. Now, Dr. Otto, if any competent engineer, familiar with (O. 59) mechanical arrangements and treatment of gases, were told to remove ammonia from coke oven gas by spraying a suitable liquor through the gas, you would expect him to be able to design an apparatus that would do that, without calling on anything more than engineering skill, would you not? A. No, I wouldn't—well, I tell you this: there you come to something like a gentleman I know. He said, "Well, I don't value your patents or inventions so high. I think a much higher thing than the patent is the task which you say; and some additional, because very often you can then say that you came to do it like that."

(The last answer was read.)

A. (Beginning) Let me express this: you have to solve a problem, and to put up this problem is sometimes much more difficult than to solve it, because people in this way say, "I want to divide my mind up now, to get with this liquor the ammonia out."

You see, after I had decided that that would work, then it is very easy for an engineer then to draw up

the apparatus how to do it. Do you see that—as soon as I tell him that will work, but nobody assumes that it will work because even I know that people even called me: I was absolutely crazy to go in on a thing like that, when we built the saturator in Lackawanna. And perhaps only the fact that more or less, you see, I was the only one to make the decision, I got (O. 60) it through, also, so easy.

Q. I think you answered my question with a sentence four or five sentences back, when you said that once an engineer was told that he could remove ammonia from coke oven gas by spraying a liquid through the gas, the making of the apparatus would be just an engineering matter that any good engineer could do—is that right? A. Could design the apparatus to do it, you see. But nobody decided on that—that it was possible to do that.

Q. Well, doesn't that mean, Doctor, that your position is that the essence of what you regard as your invention is spraying a suitable liquor through coke oven gas to remove— A. (Interposing) That's right.

Q. —to remove ammonia? A. To spray saturated liquor containing crystals through nozzles in a fineness to give enough contact, through coke oven gas.

Q. Well, I have to go back: the answer is yes. Now, the question of fineness, and so forth, is a matter that can be worked out by anybody by experiment? A. Yes.

Q. And it is based upon knowledge well known to any physical chemist, I assume—that is, the effect of fine and coarse sprays, and so on? A. Well, of course, when things are done, everything looks (O. 61) easy, especially the main inventions. You will know that, too.

Q. I am just trying to find out, Doctor, what you think is the essence of what you did that justified the patent. You understand that? A. Yes.

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(O. 62) Q. Are the pumps that are used and have been used in your spray saturators, standard pumps that were known and used (O. 63) before? A. Standard pumps.

Q. Have you had any trouble with those pumps clogging up? A. No, not with Worthington pumps. We have had bad experience with the La Bour pump, and also other people had it.

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(O. 64) Q. Of course, Monel was well known before your first— A. (Interposing) Oh, Monel is used for many purposes.

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(O. 65) Q. Do you have to kill your saturators occasionally? A. Yes; in some places we do.

Q. That is to remove accumulation of salt? A. That's right.

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(O. 75) Q. In the sketch that you made, you are now talking about the cracker pipe type? A. No. How we went over gradually to the spray type.

Q. It was an evolution, then, from one to the other? A. It was an evolution, step by step. I just want to recommend what was going on in my crazy brain—what I was thinking there. You see how it developed. You see, it didn't come just to me like a flash. You see, it was a development.

Q. It wasn't any flash of genius, then? A. No, no. Everybody builds up on existing things. Then it came out. Then I came over to this other thing here (O. 76) (indicating). Here, I had still on this—I had this installation with steam.

If you come to the second drawing, here is another drawing—see this (indicating). Then you come to this No. 2 in my first application here (indicating). You see, Mr. Brown, I came to that.

Mr. Morton: The witness refers to Figure 2 of the patent in suit.

A. (Continuing) Then I thought about, why not make that simpler and take this whole thing out? And that was the next step, and I came to this figure here. If that was done, we had still to do this in regard to Bethlehem, because they wanted to change to a cracker pipe saturator. We had, therefore, to make this 11 feet, maybe it was 12 feet—no, 11 feet—and this (indicating) 12 feet. But when we built after Lackawanna, after we knew the spray story worked, then we went over to 3 and 4 and built like scrubbers are built—cylindrical scrubbers. And that is what we stick to.

Q. This apparatus was something of an evolution of one step to another, and gradually you improved, as you thought, on the structure of the other one, and worked it on down over a period of time with mechanical changes? A. That's right.

Q. To arrive at what you thought was the best form that you had? (O. 77) A. That's right.

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(O. 78) Q. I suppose you are still changing and improving, and evolving, your mechanical arrangement of the elements? A. That's right. That's right.

Q. And are you prepared to say that this is a matter of engineering skill and knowledge? A. And then your experience, and you watch it and see where it can be improved, because we like to stay in business, you see. And the thing is this: to stay in business, you have to improve and you have to give your customers something really up to date. And, also, the competitors keep you going. That is the good thing, you see, about private enterprise, because new needs come up, and then you have to adapt yourself to it.

Q. Well, is it fair to say, Dr. Otto, that the characterizing feature of any of the saturators you have built, is the use of the spray through an unobstructed chamber, through which the gas passes? A. Yes.

Q. They have that in common? A. Oh, yes, they have that all in common.

Q. Now, leaving that feature out, as I understand it, the other mechanical elements can be arranged in a great variety of forms and positional relations to each other—is that fair to say? (O. 79) A. Yes, that's right, but all have this feature what you have just mentioned.

Q. And if you took that feature out, it wouldn't have much except just an engineering building proposition, would it? A. Well, I don't know if other features, perhaps, can also get some patents. It depends on the Patent Office, you see—other additionals which come up.

Q. What I am getting at is, your idea of what is the essential idea of all your apparatuses that you have built. A. This is a fundamental, main feature.

Q. A fundamental feature is spraying the liquor through an unobstructed chamber? A. That's right.

Q. And the rest of them can be varied, or, in some cases, eliminated or repositioned, or changed in form? A. That's right.

Q. Now, this morning, Doctor, you spoke of certain difficulties or obstacles you met in making a saturator using a spray through the gas. I think you named three of them. Can you think of any other difficulties that confronted you, and which required— A. What were the three?

Q. I think I can name them. I think one of them was a nozzle—a nonclogging spray nozzle that would be; another was washing the walls, if I remember rightly, to keep crust (O. 80) from forming on the walls. A. That means, also, the design would be right—if you mention

just to make it nice and smooth, make it round, without edges, and so on.

Q. That problem you met with structural design, avoiding corners and rough spots, so you would have a smooth passage for the spray downward through the chamber—is that right? A. That's right.

Q. And the third one was to get optimum efficiency of the use of your spray—is that right? A. That's right.

Q. Now, were there any other problems that confronted you in making the saturator that is described in your patent in suit? A. Well, I think it isn't mentioned—you see, this liquor has to be saturated, and contains crystals.

Q. Is that a difficulty that confronted you? A. Absolutely, because, you see, it was known to use undersaturated liquor. That was done by Wilputte.

Q. As I read your patent, it says that you can use undersaturated, saturated, or supersaturated liquor? A. Yes; but, you see, it's only important when you kill; it is very short time that it could be.

Q. Well, if your patent says that you can use undersaturated, saturated, and supersaturated liquor, at the spray nozzle, is (O. 81) that correct? A. The spray nozzle takes it, but to use undersaturated liquor, that is known. Wilputte has done that already.

Q. I am talking about you and your apparatus now. Do you have to have saturated— A. (Interposing) No.

Q. Do you have to have supersaturated liquor at the nozzle? A. No, not at the nozzle, I don't have that.

Q. Do you have to have undersaturated liquor at the nozzle? A. No, but it can deal with it. You see, my invention goes on. It is advantageous to have conditions of a saturated liquor, just as you had, also, before that, a cracker pipe saturator. You see, when you have undersaturated liquor, then you have to have a special apparatus, an evaporator, to make a saturated and supersaturated possible performance.

Q. But, I understand you to say now, that the liquor, as it comes from the sprays in your saturator, can be either unsaturated, saturated, or supersaturated—is that right? A. It can be. But the saturator works.

Q. But the feature of the patent is that it is saturated, and gets supersaturated? I think it is also expressed in the claims. A. Undersaturated is not a feature of my patent.

Q. But it can be used? A. It can be used, but it is not a feature of my patent. (O. 82) You see, if anybody else used undersaturated solution, I would say that he saw my patent.

Q. Well, Doctor, as I understand it, you state as your position that your process, embodying or utilizing the invention claimed in your patent, can start at the spray nozzles with (1) unsaturated liquor, (2) saturated liquor, or (3) supersaturated liquor. Am I right or wrong about that? A. Oh, no. I say I usually use saturated liquor and get it supersaturated, and then let it get desupersaturated in some vessels.

Q. Well, I am talking about at the time it leaves the spray nozzles. A. Then it gets supersaturated.

Q. All right. But, suppose it starts out unsaturated? A. But it also begins right away, because crystals are present; the desupersaturation also begins right away.

Q. But, it is all right for the supersaturation, or the saturation, to appear after the spray is passed from the nozzle and passes down through the chamber—is that right? A. Yes. But always crystals are present there.

Q. Well, now, those crystals that are present in the liquor that sprayed through your gas chamber, are crystals that have been formed in the liquor that has passed down through the chamber in the form of spray—is that not right? A. That are additional crystals—that are additional crystals, (O. 83) also, you see. There are crystals present in the liquor already before it is sprayed.

Q. Aren't those crystals there because it has already been passed through— A. (Interposing) It is from before, because it is always circulated.

(The last question was read.)

Q. That is where you get your crystals, from the liquor that is sprayed? A. It starts.

Q. And do you say the passage of those crystals is essential to your process? A. Essential to our patent application because they are in it, because, you see, just like in a cracker pipe saturator you have also the crystals present.

Q. Where do you get the crystals that are in the liquor that you spray in the process of your patent? Where do they get into the liquor? A. They are in the container from where the liquor is pumped.

Q. Well, they are there, inevitably, aren't they, where you recirculate the liquor? A. I recirculate.

Q. You couldn't get them out if you wanted to? A. You could get them out if you made them very big, these vessels.

(O. 84) Q. It would be very expensive, wouldn't it? A. It would be very expensive and impractical. Not necessary to do that.

Q. So, as I understand it, the presence of small crystals in the liquor that you spray down through the gas chamber, is what might be said to be the practically unavoidable result of previous use of the liquor—isn't that right? A. That's right.

Q. You don't actually put them in, intentionally, do you? A. No, no. I don't put them in, but they develop, because it is the idea to make crystals.

Q. Can you keep them from developing where you recirculate liquor? A. You could, for example, sometimes destroy them. That is done in liquor coming from the

dryers. It can be steamed. You can destroy them with steam.

Q. But that would be an expensive and impractical— A. (Interposing) Well, of course. For example, in this Wilputte place, in Lorain, they do it in this big bath. They destroy the crystals. They want to make especially big crystals, so they destroy the amount of seed crystals. It is something like birth control—you destroy and kill the babies.

Q. What I am getting at, you don't intentionally, purposely, provide any means for securing seed crystals in the liquor (O. 85) that you spray down through your— A. (Interposing) No, not purposely.

Q. And to prevent that would require an impractical and expensive process? A. That's right. A process which has worked, economically worked, was the old cracker pipe saturator, and I want to keep up, as far as possible, these conditions. You see, in the cracker pipe saturator, you have also this.

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(O. 86) Q. What I am asking you is—and it is the only point that I had in mind—there is no virtue in one spray as distinguished from two sprays or half a dozen sprays, or whether they are arranged around in a circle, or whether there is one in the middle that covers the whole chamber? A. Mr. Brown, I would only say this: if you make too many sprays, then the sprays are so small, then you come to a point where the sprays will block up. I couldn't tell you where that point is. Then you find out, according, where you would have difficulties.

Q. Then, you reduce it to an absurdity? A. That's right.

Q. So, you can make any arrangement of sprays, with a wide variety, a wide range of variations, and still get satisfactory results? A. That's right.

Q. And, also, you have shown some sprays for washing down the walls. You could use them or not use them, could you not? A. Yes. That means, for example, if we don't use it, we have to put more killing and more water in

(O. 87) Q. You would have to kill it more often? A. That's right. And clean it, and so on.

Q. In other words, it is not the essence of your invention, it is simply a matter of good engineering and choice, according to what you want to do in a particular case—is that right? A. That's right, yes, sir.

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(O. 88) Q. And if we were to show you a drawing dated prior to your date of invention, which, as I recall, is in 1946—your invention, as you conceive it to be—which showed a spray at the top of a cylindrical chamber through which coke oven gas was being passed, to be treated by the spray, and the liquor resulting from the spray was collected in the bottom of that chamber, that would meet the essential elements of your patented invention, as you understand it, in so far as treating gas—coke oven gas—with a spray, is concerned, would it not? A. I don't know. But I didn't see such a construction yet.

Mr. Brown: I will have the question read, if you want. I am just asking you a simple question. I will (O. 89) have it read if you want, because I think you can answer it without seeing what I am talking about. If you can't answer, then, of course, that is out. But I think you can answer it.

(The last question was read.)

A. You say you have a drawing?

Q. No, I didn't say that. I said, if I were to show you such a drawing. A. If you would show me such a drawing?

Q. Yes. A. Yes, of course, if you have such a drawing which reads to this (indicating), and, of course, I can see that it is the same, isn't it? Is it patented already—if you find the construction even the same, and described the same?

(The question that was previously read, was read again.)

A. I wouldn't say that. No, I wouldn't say that, because you have mentioned a patent back of it.

Q. I don't think I mentioned any patent, sir. A. You mentioned a patent back of it.

Q. Now, Doctor, I am asking you, as an engineer and as a patentee, and as a builder of saturators and as an engineer who designed a great many saturators, what seemed to me to be a rather simple question. You will note that the question left out the matter of recirculation of liquor containing (O. 90) crystals. But, I am asking you as to the element of treating coke oven gas with a spray and collecting the liquor in the bottom of the chamber through which the gas and the spray passed, if that would not meet that particular feature of your invention, as you understand it. A. It would meet, to have just a spray there, and not a circulation, and not to talk about what kind of liquor you are circulating. It refers just that there is a spray.

Q. It would meet the spray part of it? A. Yes, sir, it would meet the spray.

Q. Well, now, if that drawing or whatever text went with it, showed that liquor was drawn off from the bottom of that chamber, and recirculated to the sprays at the top, it would inevitably have entrained crystals in the spray, would it not? A. I wouldn't say that. Did that say it had?

Q. Let us assume that it just took the liquor out of the bottom and at least took part of it up to the top and put it down again through the spray. The spray would have entrained crystals, would it not? A. Under certain conditions, yes; under certain conditions; but you have to make the conditions right.

Q. Well, I will assume that it hasn't been put through evaporators, and so forth. A. You see, if others had these ideas, they didn't go ahead and build it on a big scale.

(O. 91) Mr. Brown: Let us not argue the case now. Mr. Morton will do that for you.

Q. All I am trying to get you to tell me— A. (Interposing) That you know yourself, I think.

Q. Sir? A. That you can answer yourself. If that is the case, well, then you have it.

Q. Then, that is that? A. Yes, but I think that is common sense.

Q. Don't you know of any patent or publication, prior to 1946, which showed and described apparatus in which coke oven gas was passed through a chamber at the top of which sprays were located, and the sprays were supplied from liquor which was drawn from the bottom of that chamber? A. I only know what you have mentioned to us—this patent of Becker that you referred to.

Q. I didn't mention a patent of Becker. I am quite sure I didn't. A. I am sure you did. You brought it up.

Q. Does your answer mean that Becker shows that arrangement—this combination that I have stated in my question to you? And remember, you brought Becker into it; I didn't. A. No; I just wonder if that is it. Is this it (indicating)?

Q. Now, my question was—I asked you a question, and, as I understood you to say, in effect, that is what Becker shows. (O. 92) Is that what you say Becker shows? A. Well,

it seems that you say that this is just the same. That is how I understand that you refer to this patent.

Q. I don't accept that. I didn't say that I was referring to anything. I was asking you. A. You mentioned several patents to us.

Q. I may have, but I don't remember mentioning any patents except those that you mentioned.

Mr. Morton: I think that what the witness had in mind by saying that you mentioned it, was that the answer recited certain patents.

The Witness: You cited certain patents against us.

Q. Now, understand, I didn't ask you about the Becker patent, but I thought you said that that was—that my question was, in effect, a statement of what Becker shows. Is that what you mean to say? A. Yes, that is what I think.

Q. That is what Becker shows? Becker shows what I asked you about—is that right? A. That's what I think there.

Mr. Brown: Mr. Morton, Dr. Otto has pulled a patent out, and you and he are looking at it. Would you mind giving us the number and date?

Mr. Morton: 1,307,534.

Q. Do you know, Dr. Otto, whether or not Mr. Underwood had (O. 93) put sprays in a cracker-type saturator used by Bethlehem prior to 1946? A. No, I didn't know.

Q. Did you ever hear that he had done that? A. No. You told me that.

Q. Sir?. A. I hear it now. I assume that he did it.

Q. I didn't say that. I asked you. A. Yes.

Q. But you never heard of his having done it? A. No, I never heard it.

Q. Now, this agitation of the crystallizing pool or bath where the crystals are being formed, do you regard it as new with you—the first to discover that you could vary the size of the crystals formed by agitating the liquor? A. No. Agitation—that was well known by the cracker pipe saturators. The cracker pipe saturators all agitate to make bigger crystals.

Q. And they can control, to a large extent, the size of the crystals— A. (Interposing) That is not new.

Q. —by the rate of agitation? A. That is just the same as people—you express it more scientifically, you call it a more stable condition—that you don't oversaturate it. It's all the same principle, which is (O. 94) very old and very well known. I have known that, I will say, forty years ago.

Q. And this feature of agitating the liquor in the crystallizing chamber in various ways, as, for example, forcing the liquor to flow upward in it, or pumping it in the side— A. (Interposing) You can do it in the pump. You can have a propeller in it. You can do it by air. You can do it by gas. You see, there are many possibilities.

Q. That was old matter, and you don't claim that in any respect as a part of your invention? A. No.

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(O. 95) Q. Now, your patent has a good deal about the growth of the seed crystals as they pass down in the spray and as they exist in the crystallizing chamber, and about the production of crystals of various sizes, according to what they are to be used for. That was well known before, wasn't it? A. Oh, yes. You have that with cracker pipe saturators, and with evaporators you make it.

Q. And, of course, it was old in the art to take the tar out of it? A. Oh, yes, very old.

Q. I think you said this morning that it was old to use acid removers? A. Acid removers? Oh, I know what you mean—acid catchers. You said acid removers.

Q. I am sorry. I should have been more explicit. That was old stuff? (O. 96) A. Oh, yes, you have many, but still you have patents. Wilputte got one to use their crystallizers in the top.

Q. Now, the size of crystal that you want to get, of the ammonium sulfate, that depends upon what it is to be sold and used for, does it not? A. That's right. It depends on the condition of the market. You see, the cheapest is to make very small crystals.

Q. The cheapest would be the small— A. (Interposing) The cheapest crystals are the smallest.

Q. But, as I understand it, for fertilizer, and some things, they want larger crystals? A. When they throw it out by hand, they want larger crystals, especially for China. They want the especially large crystals for export. But, in America, it's about 70, 80 per cent of all the salt is dissolved again.

Q. And if your crystals are too small, they would tend to cake, would they not? A. That's right. There is no flowing, and there is more trouble in handling it.

Q. For certain purposes, you want them to flow like salt, or rice, out of a sack? A. That's right.

Q. For others, they might want it in cake? A. That's right.

Q. And you regulate your circulation, and so forth, to give (O. 97) the people who are buying the crystals what they want? A. That's right. That is especially done—you find that in Europe.

Q. That is just a normal way of making crystals, which was old in this art, and other arts, I assume? A. That's right.

Q. Now, does it make any particular difference, so far as utilizing the method claimed in your patent in suit, where you add sulfuric acid? A. You mean in regard to the process where I add the sulfuric acid?

Q. Yes. I mean, you can add the sulfuric acid at a number of different places? A. Yes, you can. For example, for taking the ammonia out, you see, the ammonia you take out if you put it in, in any place, but, of course, you take it better out when you take it out—when you make the last spray higher in acidity.

Q. And, if you wanted to have crystals free from sulfuric acid, you might put it in at a different place? A. That's right. You see, my plans—I have shown there that you can put it in in the last spray. It is the highest in acidity. And I have also shown you in 3 and 4, we have only one pump. Of course, then, it's all the same.

Q. That is what I had in mind, and I wanted to make sure that you didn't claim anything particularly about prior— (O. 98) A. (Interposing) Some claims claim that, and others not. There are some claims in the higher acidity in the last spray. I have a claim, you see, where I have higher acidity in the last spray. I think you will have noticed that.

Q. Won't you admit that it is perfectly obvious that the result of putting in the sulfuric acid would affect your end products, so to speak, of your process? You might want to put it in one place to get one result, and another place to get another result? A. That's right. For example, where the conditions are—if you want to take something out. But it is especially difficult if you want to cut down very low, you see. Where you are low in the ammonia content, it is very advisable to have the last spray very high. Also, you take the impurities out. Also, impurities is a weaker base. You take it out easier. It's like you take out the last traces of ammonia, which is more difficult. If you take any base out which is more difficult to take out, then it is important to have a higher acid content, especially at the last spray.

Q. I think I understand that perfectly. I just want to ask you one other thing about it, and that is, wouldn't

any chemical engineer know what would result by putting the acid in at one place, as distinguished from another? A. You mean, if any chemical engineer would say what kind of a difference?

Q. Yes. Couldn't any chemical engineer tell you, or couldn't (O. 99) he have told you in 1946, what the result would be of putting the acid in, say, at one place, as distinguished from putting it in at another place? A. If I ask him just this question, if I put it in there or there, we will say, there is this advantage and there is that advantage.

Q. But, wouldn't he be able to tell you what the result would be if you told him what you wanted him to do with it? A. Yes, but, still, you have to suit your purpose, and, therefore, it is important that you provide that.

Q. But, having chosen your purpose, then it would be an obvious thing as to where you would add your acid, isn't that right? A. That is something which is also in connection with my spray saturator, because, you see, with the saturator that you had before, the cracker pipe saturator, it was practically not possible to bring later on, the gas, with the cracker pipe, in content with the higher acidity. You could do this, so I think you can argue, also, in favor—that that is an improvement. I just tell you that I can argue with you, that.

Q. Of course, I am leaving out the cracker pipe variety because, as you say, maybe you couldn't put it in. But, your apparatus is such that you could put it in in a variety of places. The point I wanted to get, I think you answered, but I will ask you again: anybody conversant with the art could (O. 100) have told you what the difference would be of adding the sulfuric acid at the different places where you have added it? A. I tell you this: if you once made this step and found out that this worked, then you could see this is another advantage.

I just like to say I think there is still another advantage in that.

Q. Your point is that, with the apparatus shown in the patent, it has the advantage that you could add the acid at different places for different purposes—is that right? A. That's right. Well, look here. May I just say this. I hope you don't mind. I know, in Lackawanna—once we are told by the people in Lackawanna that the Koppers Company only has the last spray—when you copied it—the higher acidity. Then, they pointed out, and said, "Well, you see, we have done that already, here." So, I think, at that time, you see, if you sit on the other side of the table, you would argue differently. I would just like to say that.

Q. Well, Doctor, what I am trying to get at is, the place where you add acid in the apparatus shown in your patent— A. (Interposing) Yes.

Q. —is not critical. But that by adding it in different places, you can get different desirable or undesirable results? A. For different purposes, that's right: that's right.

Q. And the thing that I wanted to ask you was whether or not, once an apparatus was made so you could add it at (O. 101) different points, any chemical engineer could have told you what would be the result, could he not? A. Yes, he could. Just like when you have made an invention, then you can say that.

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(O. 102) Q. Dr. Otto, does your information permit you to say whether or not the Wheeling saturators that are here involved utilize the liquid seal, such as element 61 of Figure 4 of your patent? A. It is a seal, but it is not the same kind of a seal.

Q. It doesn't use any "U" shaped tube extending outside? A. No. Here is the pipe going down in a container, just as you find with the Wilputte construction.

Q. The answer is no, to my question, isn't it? A. No, it is not the same. It is not the same. It's the seal.

Q. But your seal is a pipe which extends from the bottom of (O. 103) your axial chamber through the "U" shaped pipe that overflows into your crystallizer? A. That's right.

Q. And that, you understand, Wheeling does not use? A. That's right.

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(O. 108) Q. Is it fair to say that the use of a spray to treat a gas is for the purpose of getting greater contact between liquid and gas? A. That was not the first purpose. The first purpose was to get the pressure difference down.

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(O. 110) Q. If, in the cracker pipe type of saturator for treating coke oven gas to remove ammonia, sprays were placed around the cracker pipe at a substantial distance from the surface of the liquid, surrounding the lower end of the cracker pipe, would you regard those sprays as being used to treat the coke oven gas in an unobstructed space? A. If you have a cracker pipe saturator and put it first to the cracker pipe and let the bubble through, I understand, and then it comes in a space where it is unobstructed, I tell you this: there I have to ask a patent lawyer, either you or Mr. Hubbell, or Mr. Morton, if that would still be unobstructed space. I couldn't say that. You see, you can argue there.

Q. You see, Dr. Otto, your patent, in a number of places, and the claim, speaks of the gas being treated by a spray in an unobstructed space. What I want to get from you is what you regard as being an unobstructed space. As I understood you yesterday, you said it was a space in which there were no baffles or transverse partitions of that kind. A. It is an empty space.

Q. An empty space? A. An empty space.

Q. Now, if you had a cracker pipe, which, I assume, would be at the axis of a cylindrical tank, then there would be a (O. 111) space between the cracker pipe and the outer casing, or shell, that is annular space, you understand, and if the spray was placed at the top of that annular space, would you regard that spray as being in an unobstructed space? A. If you take it alone, you can argue it, but you can also argue it the other way.

Q. You can't argue with yourself. Now, how do you resolve it from your own point of view? A. You see, first, I would never do it. Either I would put in a cracker pipe, but I won't put it in a spray surrounding—

Q. You have not answered the question. A. You want me to say yes or no? It is very difficult, because, you see, you can say obstructed because you can say the cracker pipe itself does not make it empty any more, because the space is not empty, there is a cracker pipe in it.

Q. Well, it is empty around the cracker pipe? A. That's right, around the cracker pipe.

Q. Isn't the space between the cracker pipe and the shell an unobstructed space? A. You can argue this way and this way, I can tell you that.

Q. Well, you argue it out with yourself, and give me the answer you want to make to that. A. I will tell you this, because that is—you can argue that because there is still the cracker pipe in it, you see. In an unobstructed space, I understand, of course, that it is (O. 112) entirely empty, that you only work with sprays and take it out there, not with the cracker pipe.

Q. Well, now, the cracker pipe in the installation that we are talking about, where you have the cylindrical shell and the cracker pipe of less diameter—with an annular space around it, the cracker pipe wall won't be any more obstructive than the walls of the shell itself, would it? A. Well, you can use that, but then you do nothing in my in-

vention any more; then you add a cracker pipe. This is, of course, an obstruction, you can say that.

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(O. 113) Q. Dr. Otto, when you first went up to Bethlehem Steel Company, did you find that that company was using sprays to treat gas in unobstructed spaces for absorbing light oil such as benzol, tuluol, and so forth, from coke oven gas by spraying a wash oil into the benzol scrubbers? A. You ask me the question, when I first came to Bethlehem—(O. 114) first contact?

Q. That's right. A. That is not right. When I first came to Bethlehem I didn't hear anything about the pipe in the plant. I only was such an optimist, I thought I could sell a coke oven, and I talked with them about coke oven construction at that time. That later developed, after we got this order, then I heard that they had benzol scrubbers where they sprayed in liquor in an unobstructed shell, that's right, for benzol absorption.

Q. And that use of sprays in unobstructed spaces was in use by Bethlehem before you installed a spray-type saturator there, was it not? A. That's right.

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(O. 116) Q. Doctor, did you say that you knew about the benzol scrubbers, about which I interrogated you before? A. Yes.

Q. When you first went to Bethlehem—that is, you knew they were so using sprays at that time? A. That's right.

Q. And that was before you had made any spray-type ammonia saturators for coke oven gas? A. That's right: it was.

Q. I asked you yesterday, as I recall, if you learned when you first went to Bethlehem, where Mr. Underwood was, at that (O. 117) plant, that they had been using sprays to inject the liquor we are talking about into their satu-

rator chambers of the cracker type, and I think I tied the question to Mr. Underwood. Now, aside from Mr. Underwood, did you learn when you first went up there, that Bethlehem had previously put sprays into their saturator cracker-type installations, and had used those sprays in addition to the cracker tubes? A. No, I didn't know that.

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(O. 118) Q. Dr. Otto, as I recall your testimony of yesterday, and also your testimony this morning, you have testified that you have no knowledge, even up to the present time, of the use of sprays to remove ammonia from coke oven gas in the old cracker pipe saturators at the Bethlehem, Pennsylvania plant, of the Bethlehem Steel Company —is that correct? A. No. That is correct.

Q. In other words, my understanding is correct? A. Yes, in regard to removing ammonia from the gas at the saturators, in the Bethlehem Steel plant, with sprays.

Q. They didn't use sprays? A. No.

Q. In any way? A. But I assume it now.

Q. What is that? A. But I assume it now.

Q. Up to the present time you have had no such knowledge, and the only knowledge you have is what you infer from my question? A. That's right.

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Deposition of James A. Beatty.

(B. 4) James A. Beatty, a witness called on behalf of the defendants, being duly sworn, was examined and testified as follows on direct examination by Mr. Brown: This testimony was offered in evidence by the defendants.

Q. Your full name is James A. Beatty? A. Yes, sir.

Q. And your residence is Bethlehem, Pennsylvania? A. Yes, sir.

Q. And I believe you are employed by the Bethlehem Steel Company of Bethlehem, Pennsylvania, is that right? A. Yes.

Q. What is your position with the Bethlehem Steel Company? A. Superintendent of the coke oven division.

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(B. 5) Q. Have you been continuously employed by Bethlehem Steel Company during the period since 1917? A. Yes, sir.

Q. And your capacity has been in connection with the coke oven division of the Bethlehem Steel Company? A. I have been with the coke oven division since I have been here.

Q. Are you familiar with the coke ovens of Bethlehem Steel Company at Bethlehem, and particularly with the saturators used with the coke ovens for removing ammonia gas from coke oven gas? A. I am familiar with the saturators of the Bethlehem Steel Company, Bethlehem, Pennsylvania, the present saturators.

Q. Now, I believe that in or about 1917, the Koppers Company of Pittsburgh installed a battery of saturators in connection with your coke ovens for removing ammonia gas from the coke oven gas, is that correct? A. They might have been constructed in 1917; I think they went into operation in 1918; they might have been constructed in 1917, but it was in 1918 that they went into operation. A battery, you said—five or six.

Q. I believe I said five. (B. 6) A. Five is correct.

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(B. 14) Q. Mr. Beatty, do you recall how the level of the liquor in the bottom of the tanks surrounding the cracker pipes [in the five 1918 saturators] was measured or determined? In other words, was there a gauge or float or something? A. If I remember correctly, it was a gas pres-

sure gauge, showing the gas pressure, if I remember correctly, at about 30 inches. That is what we tried to carry it at, a 30-inch differential.

Q. By that I understand that if the pressure dropped, you knew that the liquor level had dropped? A. Yes.

Q. And therefore less pressure was required to force the gas through the cracker pipe, is that right? A. That's right, so we tried to maintain around 30 inches under normal operations for good operation.

Q. And that I assume was done by a pressure measuring instrument applied to the gas line as it was supplied to the saturator? A. The inlet and output. What I mean, the difference between the two pressures.

Q. The difference between the inlet and outlet? (15) A. That's right.

* * * * *

(B. 20) Q. Mr. Beatty, do you remember a change made in the [1918] saturators shown on the drawings that have been identified, consisting in putting inside the saturator tanks means for discharging the sulphuric acid-bearing liquor through the coke oven gas in the form of sprays for spraying the liquor? A. We made changes in the saturators, but to the best of my knowledge, we never had any sprays; we had holes in pipes there, but I wouldn't call them sprays.

Q. Do you have drawings showing this change made in these saturators? A. Yes, sir.

* * * * *

(B. 29) Cross Examination by Mr. Morton:

Q. Do you recall, Mr. Beatty, that the changes were made at or about the dates that are shown on these drawings [showing the Underwood changes to the 1918 Koppers saturators, also shown on DX 14], that is to say,

1944 or thereabouts? A. Well, approximately. These were first put in operation in September, 1943, approximately; I don't know whether that is the exact date, but approximately September, 1943, and according to the drawings there were different changes made after that time, and this operation was discontinued in June, in approximately June of 1944.

Q. What type of operation succeeded the one which was discontinued in June of 1944? A. All these here changes were torn out and the saturators were put back in their normal condition, their previous condition.

Q. That is to say, substantially in the condition shown in this [1917] Koppers drawing? * * * A. Yes, sir, and these saturators were completely put out of operation on November 11, 1946; that is when they were discontinued entirely.

Q. What sort of a saturator succeeded the saturators (B. 30) we have been discussing? A. On November 11, 1946, the two Otto Cracker Pipe Type saturators were installed; if there is another name for them, I don't know. That is what we always called them, the Otto Cracker Pipe, 10 feet six inch-diameter saturators, cracker pipe.

Q. You have stated that you are familiar with the present saturators now in operation at Bethlehem; are those the same Otto Cracker Pipe saturators? A. No; on June 23, 1953, the Otto Cracker Pipe saturators were shut down, and the Otto Spray Type saturator was put in operation.

Q. And that is just a single saturator? A. A single saturator; that was on June 23, 1953.

Q. Can you tell me whether the volume of gas that was handled by the five saturators of the type shown in these Koppers drawings that we have been discussing remained the same for the two Otto Cracker Pipe saturators, supplemented by the one Otto Spray saturator? A. Well, approximately the same. With the five saturators, the same

amount of gas went through as with the two Cracker Type, but there is an increase in gas. With the five saturators, there was approximately 70,000,000 a day, and 70,000,000 a day went through the two first Cracker Type saturators.

(B. 31) Q. How much is now going through? A. Now going through, we added another battery, and we get more volume with the one spray-type saturator; we get about 95,000,000 through the one spray-type saturator that was put in operation on June 23, 1953.

Re-direct Examination by Mr. Brown:

* * * * *

(B. 33) Q. Do you know of your own knowledge, whether or not it was Mr. Underwood who designed and put in the changes in the 1918 saturators that have been shown by [the drawings corresponding essentially to DX 14]?

* * * * *

(B. 34) A. To the best of my knowledge, these are all Mr. Underwood's ideas; he always had a lot of ideas, and this is one of them.

Q. Do you know whether or not he advocated the use of sprays or other means of injecting the liquid through the gas in these saturators? A. As far as I remember, why, there was never anything mentioned like a spray saturator, due to the large pressure in the saturator. He was trying to manipulate around to try to drop the pressure in the saturator. As to a spray saturator, I don't think he ever had it in his mind or anything of that sort.

Q. I am only asking you your personal knowledge. A. As far as I know, he never mentioned any spray saturator to me.

Q. Now, did you ever see Mr. Underwood and Doctor Otto together in the period between 1917 and 1943, so far as you can recall? A. To the best of my knowledge, I never saw them together.

Q. Did you ever see them together at any time? A. They were together maybe five or six months before this saturator was put in operation; some time in 1945 or some place around there.

Q. It is your recollection that you saw Doctor Otto (B. 33) and Mr. Underwood together in about 1945? A. Some place around that neighborhood; it might have been late 1944 or early 1945, some place in that neighborhood.

Q. Did you sit in any conference between Mr. Underwood and Doctor Otto? A. In regards to what?

Q. In regard to saturators for removing ammonia from coke oven gas. A. I sat in a conference with them when we were talking about this new cracker type Otto saturator that we put in operation on November 11, 1946.

Q. That was the cracker pipe type installation? A. The cracker pipe saturator, yes, sir.

Q. Do you recall whether Doctor Otto at that time recommended the cracker pipe type of installation for Bethlehem use? A. I believe so, because that is what they presented in their conference, and that is what they were trying to sell us, so I guess they would recommend this cracker pipe type saturator at that time; in fact, that is what they recommended to us; that is the only thing they did recommend to us.

Q. That might have been in 1945? A. Well, it might have been the latter part of 1945 or it might have been the early part of 1946. I just don't remember those dates. I wouldn't want to state what the dates (B. 36) were. It was some time before November 11, 1946; it might have been six or eight or nine months, maybe a little longer than that, because we figure on a new saturator, it takes a while to get them constructed, so it might have been eight or nine months, maybe a year.

Q. But it might have been less? A. It might have been less, I wouldn't want to say.

* * * * *

Deposition of William J. Toohey, Jr.

(B. 55) WILLIAM J. TOOHEY, JR., a witness called on behalf of the defendants, being duly sworn, was examined and testified as follows: This testimony was offered in evidence by the defendants.

Direct Examination by Mr. Brown:

Q. What is your full name? A. William J. Toohey.

Q. Will you please state your residence? A. Bethlehem, Pennsylvania.

* * * * *

(B. 56) Q. In what capacity were you employed by the Bethlehem Steel Company of Pennsylvania from the time when you began work for them on up through the time you were employed by them? A. Would you like a complete history of my various positions?

(B. 57) Q. Yes, I would, if you would do so; it will probably save time. A. Well, I was employed as a looper, and worked as a looper until October, 1941; from 1941, October, until December, 1943, I was a metallurgical observer, and from that period on I was chemical engineer, coke oven division.

Q. From that period on means from what date? A. From December, 1943 until November of 1954; from that period on I was employed and am employed in the sales department.

Q. Did your work at the Bethlehem Steel Company plant at Bethlehem, Pennsylvania, during the years, say, from 1940 through 1946, bring you into contact with the saturators at Bethlehem, used for removing ammonia gas from coke oven gas? A. During the period from October, 1941 through 1946, yes, sir.

Q. Were you familiar with the structure and operation of the saturators that have been stated by Mr. Beatty and

Mr. Reilly as having been installed by Koppers Company in 1917? A. Yes.

* * * * *

(B. 58) Q. In the years 1942 and 1943, did you know that the saturators as built by Koppers in 1917 or 1918 had been changed by incorporating the structure shown on drawing X-3942 [DX 11]? A. During the period that you mention I knew the saturators had been changed, and within the last few months I learned they were changed to incorporate the structure as shown on this drawing that you mention here.

Q. In the years 1942, 1943, 1944 and 1945, did you have occasion to observe the operation of the saturators shown on drawing X-3942 [DX 11]? A. What do you mean, observe?

Q. Well, did you see them and did you have anything to do with checking the effectiveness of operation of those saturators during that period? A. I saw them operating, but had nothing to do with the actual operation nor anything to do with checking the operation of those saturators.

* * * * *

(B. 59) Q. Before you saw these drawings, did you know that the old Koppers 1918 installation had been modified to put in the spray pipes which I will mark with the letter "H" on drawing X-3942 [DX 11]? A. I knew nothing of the internal changes that were made, other than Mr. Underwood and Mr. Young were experimenting with a low differenital saturator. As to the changes that were made, I knew nothing of them, the internal changes to the saturator.

Q. Did you know that Mr. Young and Mr. Underwood were experimenting with the use of sprays in addition to cracker pipes in these saturators? A. You say they were experimenting with the use of sprays?

Q. I am using your word "experimenting"; I don't

mean that to be technical. A. I was merely attempting to explain, by using that language, that I knew they were experimenting with a low differential saturator; I never specifically heard them refer to this as a spray experiment or a spray saturator.

(B. 60) Q. Well, did you know that they re-circulated the sulphuric acid liquor so as to introduce it into the gas chamber by pipes other than the cracker pipe? A. Yes.

Q. You knew that? A. Yes, sir.

Q. When did you know that, first? A. During the period when they were running this test.

Q. And that would be in 1942 or 1943 or 1944? A. According to our recent check of the period here, I think it was during the period of late 1944 or early 1945; I can't just from memory be sure; it may have been late 1943 or early 1944; I am not sure.

Q. Did you sit in on any meetings at which Mr. Underwood and/or Mr. Young was present when they discussed the lowering of the differential pressure of the gas before it entered and after it passed out of the saturator by spraying the acid liquor into the body of the gas as distinguished from passing the gas through the cracker pipes into the body of the liquid? A. I have no specific recollection of sitting in at such a meeting. I can assume, that I was present at such meetings, since we had meetings three times a week in which we discussed all phases of plant operation, and I would assume this problem came up in the course of one of those meetings.

(B. 61) Q. Did you discuss with Mr. Underwood and/or Mr. Young the matter of the possibility of lowering the differential in pressure of the coke oven gas before and after it passed through the saturator by using sprays in the saturator chamber? A. No, I did not.

Q. Did you ever hear Mr. Underwood speak of using sprays as distinguished from cracker pipes for removing

the ammonia from the coke oven gas? A. I have no specific recollection of hearing Mr. Underwood speak of sprays.

Q. Does that apply to Mr. Young, too? A. Or Mr. Young.

Q. When did you first learn that a structure such as is shown on drawing N-3942 [DX 11] was being used in the Bethlehem saturators? A. Do you mean, when did I first learn of the actual structure itself, the details of the design?

Q. Yes. A. As I said, I knew at the time of the experiment, that the experiment was being performed, but I did not know the details of the experiment.

Q. But you knew that the broad idea of spraying the sulphuric acid liquor into the gas was being tried out, is that right? (B. 62) A. I would like to emphasize once again, never was this referred to as a spray saturator, and I never thought of it as a spray saturator. I was aware of experimenting on a low differential saturator, and at the same time I was performing other spray experiments of my own, relating to another piece of equipment, but as for thinking of this pipe, which I didn't even know existed, with these deflector sprays, which I didn't know existed, as a piece of spray equipment, would be entirely foreign to my thinking of a spray or piece of spray equipment.

* * * * *

(B. 72) Q. Assuming that liquor such as that in the sulphuric acid bath were forced by the pumps which you have pointed out, through these pipes, and if the perforations in the pipes were immediately adjacent to these curved deflectors, would you not expect the material forced through these openings in the pipes against these deflectors to take a general direction which (B. 73) might be roughly indicated by the faint lines about which we are talking? A. I would not expect it to follow that direction at all.

Q. What direction would you think it would take? A. A lot would depend upon the pump capacity, of which I know nothing, and also on the pump, of which I know nothing; the head at the discharge opening of the pipe. It could squirt out as a small stream with no velocity, and without knowing anything about the pump, I can't give you an answer to that question.

* * * * *

Q. Do you agree it would be? A. Assuming that you had enough pressure at the discharge opening, I agree there would be a spray thrown off the deflector, or a stream thrown off the deflector, thrown up into the chamber. I would expect under the circumstances that part of it would drop down, and part of it would be carried away mechanically by the gas passing through the saturator.

* * * * *

(B. 77) Q. But from your general knowledge of saturators and the chemistry involved, by which, of course, I mean solution of sulphuric acid being used to contact coke oven gas for the (B. 78) purpose of removing ammonia in the form of ammonium sulphate crystals, would you agree that causing the liquid to pass through these [Underwood] pipes under pressure and to be directed by the "spray deflectors" would cause the gas in the saturator chamber to be intimately and substantially contacted by the sulphuric acid solution? A. In this specific case, when you use the word "intimately", no, I would not agree, or substantially.

Q. What would you say a spray had to be in order to give you intimate contact of the coke oven gas and the sulphuric acid solution? A. I would say, speaking very generally, that the spray would have to be somewhat finer in nature than that which could be gotten from a deflector device of this type by intimate and substantial contact with the gas and the liquor.

Q. Would you say the question of intimate contact is a matter of degree? A. It is a matter of great importance in this particular instance where you cannot tolerate any loss of ammonia in the gas leaving the saturator.

(B. 78) Q. Do you know anything about the effectiveness of the apparatus which incorporates these [Underwood] spray pipes and deflectors in actual use; I mean of your own knowledge? A. These particular ones?

Q. Yes. (B. 79) A. I know what experiments were conducted in this prior period, and from my knowledge which I have gained within the last two months of the design of this saturator, I am now in a position to state that I now can account for the substantial loss of ammonia that occurred during the period of these tests.

Q. How do you know there was a substantial loss of ammonia during this period? A. Because our production record during the period of the test showed it.

Q. Do you have those production records? A. No, but I have a recollection of the loss. Our normal ammonium sulphate I think was in the neighborhood of 18 to 20 pounds per ton of coal charge, and I know during this period we fell down to 13 pounds of ammonium sulphate per ton of coal charge.

Q. Well, since you have been very hazy on your recollection about this apparatus and its operation, in response to other questions that I have asked you, how is it you are able to give us exact quantitative statements of the amount of ammonia that passed through these saturators when they were used with the arrangements shown in these [Underwood] drawings, * * * A. Since you are depending on 100 percent of my memory, I reserve the right to go back and check my recollection (B. 80) occasionally. I would like to say that during this period, when I was not associated with the operation of the saturators, I was in charge of keeping complete material balances of the plant, and

one of my specific duties, monthly and weekly, was to correlate the distribution of materials on a weight basis, which involved checking the raw material charged to the plant, and it was my specific duty to draw this sheet up on a weekly basis, and these figures which I indicated to you are very familiar to me.

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(B. 82) Q. Doesn't that study, what you call the ammonia loss, show you the result of putting in this spray type element and the result of taking it out? You say you were familiar with that record, were familiar with the efficiency of the plant and that you have the figures on that, and that you remember the figures. Now, I ask you, do you have the records? A. I do not have these records. They were discontinued in 1948. I don't know what the disposition was of these records, but I think they were destroyed; however, as I say, I can remember this 13-pound yield figure. I cannot specifically state the time when this was done, but I can tell you this, I can associate that with the period of these tests, for this reason, when you are keeping production figures, month after month and week after week, and that figure suddenly takes a drop, you ask questions. Whether you are concerned with the operation of the saturator or not, you begin to look for that loss, and I can recall during this period our ammonium sulphate took this drop to 13 pounds per ton, and I can associate it with the period of its operation because I can remember this is the only time in the period I kept these records that such an occurrence took place.

Q. Well, now, search your memory in this. How long (B. 83) did this loss continue; you say you made weekly reports? A. I would say it continued over a period of two to three months.

Q. And during those two to three months the saturators were operating with the apparatus shown on the draw-

jets, Defendants' Exhibits 8 to 13, inclusive, is that right? A. Are you referring to this apparatus, drawing 3942?

Q. Yes. A. I can't say that, because as I told you before, I have no knowledge at all, other than by hearsay, of its construction or operation.

Q. Don't you know that this apparatus shown on this exhibit that you have just mentioned was in operation in the Bethlehem saturators for a period of two to three years? A. If you mean I actually have seen this apparatus, have seen it operate, if you are going to accept hearsay evidence, yes, I know this apparatus, from what I have heard, was being used; that this apparatus was used during the period of this test. However, during the period, going back to the period that I talked about, no, I didn't know this apparatus was in use.

Q. How long did this so-called ammonia loss continue after you first noticed it? A. I would say from a month and a half to two months.

Q. But your recollection is that these perforated (B. 84) spray pipes were installed and used over a period of two to three years in these saturators, isn't it?' A. No.

Q. Do you think they were there only for two or three months? A. I can't say.

Q. Have you any other explanation of why the ammonia loss continued only for two or three months? A. No.

Q. Didn't you make any inquiries as to what change in operation brought about this loss of ammonia? A. Not once it came back to where it belonged. I wasn't running the coke plant. I was just a little boy down there keeping records.

Q. Then you have no knowledge whatever, as I understand your testimony, as to what in the operation of the apparatus shown on Defendants' Exhibits 8 to 13, inclusive, caused this unusual loss of ammonia, is that right? A. No, I cannot pinpoint it in any way.

Q. You never had any explanation of that given to you by anyone? A. Not that I can recall. I have a recollection, which would fall definitely into the category of hearsay, that it was associated with the lack of contact of liquor with gas, and this ammonia was passing on through with the gas. However, (B. 85) I have no way of substantiating it whatsoever.

• • • • •

(B. 86) Q. If the saturators as installed by Koppers in 1917 or 1918 were operating in 1942, 1943 and 1944 for commercial treatment of your coke oven gas, would you call that an experimental use during the period of 1942, 1943 and 1944, the original saturator design? A. If the original saturator were operated during that period, certainly not. I would not call that an experiment.

Q. And if these perforated pipes were in there and used for squirting the liquor, you know what I am speaking of, liquor? A. Yes.

Q. Into the gas chamber for a period of two or three years, would you call that experimental use? A. Yes, I would. You said for a period of two or three years?

(B. 87) Q. Yes. A. No, I wouldn't call that experimental use for a period of two or three years.

Q. In other words, it would indicate a commercial use of that apparatus that was in there during the period? A. Yes, sir.

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(B. 91) Q. Do you know whether or not the Bethlehem Steel Company, prior to 1946, used a spray apparatus for removal of benzol from coke oven gas? (B. 92) A. Yes.

Q. How long had that continued prior to 1946, to your knowledge? A. It was started on a limited basis in the fall of 1943, and by 1946 the entire plant was on spray scrubbing for removal of benzol from gas.

Q. And that arrangement sprayed the liquid through

the gas in evenly divided form for absorbing the benzol in the coke oven gas, is that right? A. That's right.

Q. That was a commercial operation during the period of 1942 to 1946? A. No, I would say it was commercial during the period from 1945—about June of 1945 on, I would rate it as a commercial installation.

Q. And prior to that time had it been on what you call an experimental basis? A. Experimental, right.

Q. And extended over quite a time before 1945, as I understand it? A. That's right.

Q. And that was known to you and to other engineers for the Bethlehem Steel Company, I assume? A. Yes, it was.

Q. And to Mr. Underwood? (B. 93) A. Yes.

Q. Mr. Underwood put it in, did he not? A. Yes, he did.

Q. How fine were these sprays that were used in the benzol absorption chamber? A. It is hard to describe fineness, but I would say that the spray that we used in that apparatus gave you one of the most finely divided sprays that was possible with low pressure facilities.

Q. And did this spray apparatus for removal of benzol from coke oven gas work satisfactorily to remove the benzol? A. It did not remove as much of the benzol as we would have liked to have removed from the gas.

Q. Are you still using the spray apparatus for removal of benzol? A. The last time I knew—did you say the spray apparatus or a spray apparatus?

Q. A spray apparatus. A. Yes, we are still using a spray apparatus.

(B. 93) Now, you are a chemical engineer, I assume? A. Yes.

Q. How long have you known, in your experience, of the use of sprays for causing liquid to pass through the gas for removal of some other gas or some elements from

the gas, as a matter of general chemical engineering? (B. 94) A. The use of sprays has always been discussed in my realm as a chemical engineer. I think it began in 1943 when we started our own experiments.

Q. When you were in engineering school, did you know that that was one way of treating gas to remove impurities or mixtures of other gases? A. I knew of systems of that type being used as an absorbing medium. My information involved no chemical contacts, such as in an ammonium sulphate saturator plant.

Q. But you knew of the removal of benzol? A. That's right.

Q. That was by a contact of a liquid and gas, was it not? A. That was by absorption, but not by chemical reaction.

Q. It was by contact of a liquid and gas, was it not? A. That's right, but there is quite a difference between the two methods.

Q. You spoke of making experiments with sprays; what were you talking about; that was early in your deposition. A. I was referring to an experiment I did in the development of a spray scrubber for removal of benzol from the coke oven gas.

Q. When were those experiments commenced? (B. 95) A. I would say somewhere in the vicinity of November 1, 1953.

Q. 1953? A. That's right—pardon me, excuse me—1943.

Q. And in that experimentation that you did in 1943, how did you experiment, with different finenesses of spray? A. Yes, I did.

Q. Did any of your experiments include the chemical reaction between the liquid and gas? A. No, they did not; purely absorption.

Q. It was purely an absorption process? A. That's right.

Q. When you commenced these experiments, you did

not originate any new thought, you used the sprays per se, for treating the gas, I assume? A. No, I didn't.

Q. You, in fact, as a chemical engineer, knew, even as of 1943, sprays were what might be called a tool in the art for removing undesirable constituents carried by gas, is that right? A. I would like to amend that by saying I knew of no commercial installation in the United States involving the use of sprays for chemical or absorption removal of anything from the gas.

Q. Where did you get the idea then to use these sprays (B. 96) in your experiments? A. I didn't get the idea; Mr. Underwood gave me the idea.

Q. Mr. Underwood was quite an advocate of the use of sprays for treating gas, was he not? A. I would say he certainly was.

Q. Did he ever tell you that he believed that by spraying the mother liquid in the ammonia saturator through the gas, he could remove a substantial part of the ammonia from the gas? A. No, he didn't.

* * * * *

Deposition of H. A. Byrns.

(W. 3) H. A. Byrns, called as a witness, being first duly sworn by me to speak the truth, the whole truth and nothing but the truth, deposes and says as follows: This testimony was offered in evidence by the plaintiff.

Direct Examination by Mr. Morton:

Q. Will you state your full name and residence, please? A. Howard Allen Byrns, 3255 Dell Road, Birmingham, Alabama.

(W. 4) Q. And what is your present occupation? A. General Superintendent of Blast Furnaces, By-Products and Transportation.

Q. For what company? A. For the Woodward Iron Company.

Q. How long have you held that position? A. Since September 1945.

* * * * *

(W. 10) Q. Now, Mr. Byrns, at or about the time of this report [PX 2A to Byrns] which we can take to have been May 1948, what installation, if any, did the Woodward Iron Company have for the production of ammonium sulphate from its Coke Oven gas? A. They had at that time the old type spinners.

Q. How did that apparatus operate? A. See, the gas went into the old type saturator—there was more than one—and bubbles up through a bath and the salt was precipitated out and was put into the old type spinner and spun with the moisture thrown off and washed and the salt collected.

Q. Now, with reference to that portion of the apparatus described as the saturator, was the gas passing through the saturator in those older installations at any point subjected to a treatment with a spray of acidulated ammonia sulphate solution? A. Not that I recall.

Q. Are those old type saturators, the May 1948 saturators, still in existence? A. Not at our plant.

Q. With what, if any, apparatus for performing the same function have they been replaced? A. New type Keppers saturator.

* * * * *

(W. 11) Q. Does that new saturator, the 1950 saturator, have an apparatus for subjecting the gas that passes through it to a spray of acidulated ammonium sulphate solution? A. Yes, it does.

* * * * * *

Q. Does it include any bath through which coke oven gas bubbles? A. I don't think so.

Q. Among the papers that you brought down today from the files of the Woodward Iron Company, do you have any drawings of any sort showing the general construction and flow of your present installation? A. Yes, we do.

• • • • •

(W. 13) [Two drawings showing a saturator installation essentially like the accused Wheeling installation shown on Defts'. Ex. 16 and Pltf's. Ex. 27 were marked "Plaintiff's Bryns' Exhibits 3-a and 3-b."]

Q. Can you tell me this: Were the three saturators that existed in 1948 replaced by a single one of the type shown on (W. 14) the drawing before you, Exhibit 3? A. Yes.

Q. And is that single saturator now handling more or less gas than was being handled in 1948? A. Handling more.

• • • • •

Deposition of C. W. Phillips.

(W. 39) C. W. PHILLIPS, called as a witness, being by me first duly sworn to speak the truth, the whole truth and nothing but the truth, deposes and says as follows: This testimony was offered in evidence by the plaintiff.

Direct Examination by Mr. Morton:

Q. Will you state your full name and residence, please? A. Claude Watson Phillips. I live at Wenonah, Alabama, Jefferson County.

Q. And your present occupation is what? A. Chief Engineer for Woodward Iron Company.

• • • • •

(W. 47) Q. At the time that the Woodward Iron Company agreed to purchase that (the Koppers spray type) saturator you were then Chief Engineer as well, were you not? A. Yes.

Q. And you approved the decision to purchase it, did you not? A. Yes.

Q. And at the time you approved that decision was the design which you then approved in your understanding new and untried? A. In my experience it was new, yes, sir.

* * * * *

(W. 52) Q. Are you sufficiently familiar with the saturator operation installed as compared with the operation of the saturators that were replaced to be able to state whether the new installation constitutes an improvement on the old?

* * * * *

(W. 53) A. My impression is that it is better, the operation is better and maintenance is certainly best with this type.

Q. Well, if you were faced today with the decision you had to make in 1948, would you consider replacing the three saturators that you had then with the type that was then installed, that is, merely replacing it with the old? A. No, sir, wouldn't even consider it.

VOL.

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No. 660

IN THE

United States Court of Appeals

FOR THE FOURTH CIRCUIT

Docket No. 7401

CARL OTTO,

Plaintiff-Appellant,

vs.

KOPPERS COMPANY, INC. and
WHEELING STEEL CORPORATION,

Defendants-Appellees.

APPEAL FROM THE UNITED STATES DISTRICT COURT FOR THE
NORTHERN DISTRICT OF WEST VIRGINIA AT WHEELING

JOINT APPENDIX, VOL. II

OPINIONS, DECREES AND EXHIBITS

W. BROWN MORTON, JR., ESQ.,
ROBERT MCKAY, ESQ.,
PENNIE, EDMONDS, MORTON,
BARROWS and TAYLOR,
New York, New York,

JOHN E. HUBBELL, ESQ.,
MYRON COHEN, ESQ.,
HUBBELL and COHEN,
New York, New York,

FRANK A. O'BRIEN, JR., ESQ.,
O'BRIEN and O'BRIEN,
Wheeling, West Virginia,

For Plaintiff-Appellant.

JO. BAILY BROWN, ESQ.,
KARL B. LUTZ, ESQ.,
BROWN, CRITCHLOW, FLICK
& PECKHAM,
Pittsburgh, Pennsylvania,

WRIGHT HUGUS, ESQ.,
SCHMIDT, HUGUS & LAAS,
Wheeling, West Virginia,

For Defendants-Appellees.

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Interrogatories.

PLAINTIFF'S INTERROGATORIES UNDER RULE 33 ADDRESSED TO DEFENDANT KOPPERS COMPANY, INC.

Now comes plaintiff Carl Otto pursuant to Rule 33, Federal Rules of Civil Procedure, and demands that defendant Koppers Company, Inc. make answer, separately and fully in writing under oath and over the signature of its officer or agent duly appointed to furnish such information pertinent thereto as is available to said defendant, to the following numbered interrogatories on or before February 18, 1955:

1. Identify all installations of apparatus for the production of ammonium sulphate from coke-oven gas built, or building, from plans prepared by or under the supervision of Koppers Company, Inc. upon which construction work was in progress at any time during the period January 1, 1945 to June 14, 1954, giving as to each such installation:

(a) the name of the owner;

(b) the location;

(c) the dates of commencement and completion;

(d) the number of saturators;

(e) the type of saturators in the installation defined as

(i) "cracker pipe",

(ii) "spray", or

(iii) other.

2. Identify any drawings or plans from which the apparatus at each installation listed in the answer to Interrogatory No. 1 was constructed, and which corres-

pond or are comparable to Koppers Company, Inc. drawings Nos. 1850-2A10 of June 8, 1951, 1850-2A19 of April 23, 1951, or 1850-2A20 of April 16, 1951, giving the date and number or other description of each said drawing.

3. For each installation listed in the answer to Interrogatory No. 1 state the services performed by Koppers Company, Inc. in connection therewith and the total monies received by Koppers Company, Inc. for said services.

4. Identify all correspondence, memoranda, drawings, plans, or other written matter passing between Koppers Company, Inc. and Pittsburgh Coke & Chemical Company during the period January 1, 1945, to January 1, 1950, relative to the installation by Koppers Company, Inc. at Pittsburgh Coke & Chemical Company of apparatus for the production of ammonium sulphate from coke-oven gas, giving the date, writer and addressee or other description thereof.

5. State the names of all individuals of Koppers Company, Inc. who orally communicated with Pittsburgh Coke & Chemical Company or personnel thereof during the priod January 1, 1945, to January 1, 1950, relative to the installation of said apparatus described in Interrogatory No. 4.

6. State the names of all individuals of Pittsburgh Coke & Chemical Company with whom the individuals listed in the answer to Interrogatory No. 5 so communicated during the period January 1, 1945, to January 1, 1950, relative to the installation of said apparatus described in Interrogatory No. 4.

7. Identify all correspondence, memoranda, drawings, plans, or other written matter passing between Koppers

Company, Inc. and Woodward Iron Company during the period January 1, 1945, to January 1, 1950, relative to the installation by Koppers Company, Inc. at Woodward Iron Company of apparatus for the production of ammonium sulphate from coke-oven gas, giving the date, writer and addressee or other description thereof.

8. State the names of all individuals of Koppers Company, Inc. who orally communicated with Woodward Iron Company or personnel thereof during the period January 1, 1945, to January 1, 1950, relative to the installation of said apparatus described in Interrogatory No. 7.

9. State the names of all individuals of Woodward Iron Company with whom the individuals listed in the answer to Interrogatory No. 8 so communicated during the period January 1, 1945, to January 1, 1950, relative to the installation of said apparatus described in Interrogatory No. 7.

* * * * *

Opinion Re Interrogatories.

BOREMAN, District Judge:

This is an action for infringement of Letters Patent No. 2,599,067 issued to plaintiff on June 3, 1952, as the inventor of the invention in production of ammonium sulphate therein described. Plaintiff is a citizen and resident of the State of New York.

Defendant Koppers Company, Inc. (hereinafter referred to as "Koppers") is a corporation duly organized and existing under the laws of Delaware, having its principal place of business in Pittsburgh, Pennsylvania, and duly licensed to do business in the State of West Virginia. De-

fendant Wheeling Steel Corporation (hereinafter referred to as "Wheeling Steel") is a corporation duly organized and existing under the laws of Delaware, having its principal place of business at Wheeling, West Virginia, and duly licensed to do business in the State of West Virginia.

The complaint alleges that Koppers maintains a regular and established place of business in Brooke County, in the Northern District of West Virginia, and has committed acts of infringement in said county; that Wheeling Steel maintains a regular and established place of business in said Brooke County and has committed acts of infringement therein; that the venue of this action is laid in the Northern District of West Virginia under Title 28 United States Code, Sections 1391(b), 1391(c) and 1400(b).

The complaint charges that Koppers, without authority from the plaintiff, has been, since June 3, 1952, and still is, infringing said Letters Patent by:

> making, using and selling by-product coke oven apparatus embodying the patented invention;
>
> using methods embracing the patented invention for the continuous production of sulphate of ammonia;
>
> actively inducing others, including WHEELING STEEL, to use said apparatus and methods of the patented invention; and
>
> selling to others, including WHEELING STEEL, apparatus for use in practicing said methods of the patented invention constituting a material part of said invention, knowing the same to be especially made or especially adapted for use in an infringement of said patent, and not a staple article or commodity of commerce suitable for substantial noninfringing use,

in the Northern District of West Virginia, as well as elsewhere within the United States, and will continue to do so unless enjoined by this Court.

Wheeling Steel is charged in the complaint with infringement in the Northern District of West Virginia by procuring Koppers to make for it apparatus embodying the patented invention; using said apparatus; and using methods embracing the patented invention for the continuous production of sulphate of ammonia.

The joint answer of Koppers and Wheeling Steel, in addition to denying infringement, under affirmative defenses, puts in issue and challenges the validity of the plaintiff's patent No. 2,599,067, alleging lack of invention, prior disclosure of the claimed invention in publications and patents, the selection and aggregation of old elements and the use of old method steps, each known to those skilled in the art before plaintiff's alleged invention, and for other reasons. Said answer, by way of counterclaim, alleges that an actual controversy exists and prays for a declaratory judgment disposing of the controversy.

The joint answer of the defendants denies that either of them has committed the acts of infringement as charged in the complaint and, answering the allegation in paragraph 8 of the complaint as to venue, says:

> "Without admitting the conclusion of law implied in this paragraph, defendants waive any issue of venue that otherwise could be raised on the complaint."

The plaintiff served pursuant to Rule 33 of Federal Rules of Civil Procedure, upon Koppers seventeen interrogatories. Koppers answered a portion of interrogatory No. 10, all of interrogatory No. 15 and filed written objections to the remainder of plaintiff's interrogatories.

In briefs submitted by Koppers and by plaintiff on the objections to plaintiff's interrogatories and in argument before the Court, it appears that the answer of Koppers sufficiently challenges the plaintiff's assertion of venue under Title 28, Section 1391(b) and 1391(c) in addition to admitting venue under Section 1400(b).

Opinion Re Interrogatories.

Under the general venue statute, Title 28, Section 1391, the cited subsections are as follows:

> "(b). A civil action, wherein jurisdiction is not founded solely on diversity of citizenship, may be brought only in the judicial district where all defendants reside, except as otherwise provided by law.
>
> "(c). A corporation may be sued in any judicial district in which it is incorporated or licensed to do business or is doing business, and such judicial district shall be regarded as the residence of such corporation for venue purposes."

Title 28, Section 1400(b), reads as follows:

> "Any civil action for patent infringement may be brought in the judicial district where the defendant resides, or where the defendant has committed acts of infringement and has a regular and established place of business."

Koppers contends, in objecting to answering interrogatories, that Section 1400(b) is the exclusive venue provision in patent infringement cases; that plaintiff is attempting to broaden the jurisdiction of the Court to cover all acts done anywhere by interrogatories directed to, and intended to prove, infringement in the United States generally and particularly beyond the limits of West Virginia, and that plaintiff should not be allowed to stand on Section 1391 for general and unlimited venue and jurisdiction, while at the same time relying on Section 1400(b), under which proof of infringement in West Virginia must be established and "under which it has been held that interrogatories as to alleged infringement outside West Virginia will not be allowed".

The court decisions on the question of venue in patent infringement cases are not unanimous. Ackerman v. Hook,

183 F. 2d 11, (C. A. 3, 1952) appears to be the first Court of Appeals decision to consider Section 1400(b). That Court reviews much of the historical background of that section, the revisor's notes in connection with the adoption of the new Title 28 in 1948 and held, in effect, that Section 1400(b) is the exclusive venue provision in patent infringement cases.

There have been no decision by the Court of Appeals for the Fourth Circuit on this point. Plaintiff cites Dalton v. Shakespeare Co., 196 F. 2d 469, (C. A. 5, 1952), Guiberson Corp. v. Garrett Oil Tools, Inc., 205 F. 2d 660 (C. A. 5, 1953) (cert. denied 346 U. S. 888, rehearing denied 346 U. S. 917), supporting the proposition that the word, "resides" in Section 1400(b) is to be given the definition of "residence", as set forth in the general venue statute, relating to corporations, Section 1391(c).

Koppers calls attention to the fact that the Circuit Court of Appeals for the Seventh Circuit has taken the opposite view and cites C-O-Two Fire Equipment Co. v. Barnes, 194 F. 2d 450 (C. A. 7, 1952), which was affirmed by the United States Supreme Court by an equally divided Court, 344 U. S. 861. Since an affirmance by an equally divided Supreme Court is not binding on the lower Federal Courts, Hertz v. Woodman, 218 U. S. 205, this Court is free to follow either the Fifth Circuit or the Seventh Circuit.

There are a number of District Court decisions to the effect that Section 1400(b) is the controlling venue section in patent cases. I cite as follows:

Pierce v. Perlite (California 1952), 110 F. Supp. 684;

Nachtman v. Jones & Laughlin (Dist. of Col. 1950), 90 F. Supp. 739;

Arkay v. Klines (Mo. 1949), 85 F. Supp. 98;

Fiseher v. Karl (N. Y. 1949), 84 F. Supp. 53;

Rava v. Westinghouse (N. Y. 1950), 90 F. Supp. 707;

Federal v. Frank Adams (N. Y. 1951), 100 F. Supp. 8;
Ronson v. Brown & Bigelow (N. Y. 1952), 104 F. Supp. 716;
Transmirra v. Magnavox (N. Y. 1953), 110 F. Supp. 676.

The Court's attention has been directed to a decision of the Honorable Sterling Hutcheson, District Judge for the Eastern District of Virginia, in the case of Manville Boiler Co., Inc. v. Columbia Boiler Co. of Pottstown, Inc., as yet unpublished, wherein the Court approved the decision of the Court of Appeals for the Fifth Circuit in Dalton v. Shakespeare Co., supra. However, Judge Walter E. Hoffman, of the United States District Court for the Eastern District of Virginia, in the case of Benjamin Clayton v. Swift & Co., decided June 8, 1955, but not yet published, cited the decision of the Seven Circuit in C-O-Two Fire Equipment Co. v. Barnes, supra, and stated:

> "The practical effect of the C-O-Two case is based upon the logical reasoning that a general venue statute must give way to a special venue statute in patent infringement cases. Stonite Products Co. v. Melvin Lloyd Co., 315 U. S. 561, 62 S. Ct. 780, 86 L. Ed. 1026. If it became necessary for this Court to so hold, it would follow the principle of the C-O-Two case as otherwise Section 1400(b) would be effectively emasculated as the same may pertain to a corporate defendant."

Since the filing of the briefs by the plaintiff and Koppers in the instant case, the Circuit Court of Appeals of the Tenth Circuit has decided the case of Ruth v. Eagle-Picher Co., 24 Law Week 2080, holding that

> "The patent venue statute as construed in Stonite Co. v. Melvin Lloyd Co., 315 U. S. 561, 52 U. S. P. Q.

507, reflected a sound policy of long standing. It was based on considerations of practicality and convenience in such litigation. A patent infringement action involves a peculiar combination of science or technology and law. In the ascertainment of the pertinent technical facts, it is important that the trial judge have first-hand visual and audible knowledge of the conditions, the environment, and the art itself and the testimony of the most competent witnesses. Practicality and convenience are best served when the case is prosecuted where the alleged acts of infringement occurred and the defendant has a regular and established place of business. An intention on the part of Congress to depart from that policy should not be lightly inferred."

That Court held that the revisor's notes to the new Judicial Code clearly indicate mere language changes in Sections 1391 and 1400 and negative any intent to effect a substantial change. Moreover, where there are two statutes upon the same subject, one special and the other general, the presumption is, in the absence of an express repeal or absolute incompatibility, that the special is intended to remain in force as an exception to the general. The rule is particularly applicable where the statute is enacted at the same time or about the same time. The Tenth Circuit further stated that its conclusion is supported by a considerable body of judicial opinion and cited Ackerman v. Hook and C-O-Two Fire Equipment Co. v. Barnes.

In Dalton v. Shakespeare Co., supra, the Court apparently gave no weight to the historical background of the special venue section in patent suits, but based its decision on the technical wording of the two sections of the statute. This Court is inclined to follow the case of C-O-Two Fire Equipment Co v. Barnes, supra, and the recently decided case of Ruth v. Eagle-Picher Co. by the Tenth Circuit

Court of Appeals. Section 1391 is a general provision, while Section 1400(b) is special and specific. This Court is of the opinion that a specific provision should control a general provision and that it is improper to disregard the historical background and the purpose of the special and specific provision of the statute fixing venue in patent infringement actions.

In view of this Court's conclusion as to venue in this action, consideration must next be given to a determination of the questions raised on the objections to plaintiff's interrogatories and, particularly, the objection that certain interrogatories would require disclosure by Koppers of possible alleged acts of infringement and suspected activities outside the State of West Virginia.

Before there could be a proper claim for damages for infringement arising from the acts of Koppers and Wheeling Steel, two issues must first be decided in favor of the plaintiff: (1) The validity of his patent; and (2) infringement by the defendants within the Northern District of West Virginia.

Plaintiff's interrogatory No. 1 would require Koppers to identify all installations of apparatus for the production of ammonium sulphate from coke-oven gas, built or building, from plans prepared by or under the supervision of Koppers, at any time from January 1, 1945, to June 14, 1954, giving as to each installation the name of the owner, the location, dates of commencement and completion, the number and type of saturators defined as "cracker pipe", "spray" or other. The plaintiff argues that all acts of possible infringement anywhere in the United States are relevant, because of unlimited jurisdiction over these defendants as "resident corporations". Plaintiff argues further that he seeks evidence bearing upon the issue of validity and the affirmative defense of lack of invention and only secondarily in aid of proof of infringement. He

says he wants to show that Koppers has embodied, in all of its new mode of construction, the heart of plaintiff's invention and, in so doing, abandoned a uniform course of construction according to other modes; that, to do this, he must be allowed to inquire concerning Koppers' activities everywhere with respect to present and past installations of coke-oven by-product apparatus. In Support of this position, plaintiff cites the following cases:

Ackermans v. General Motors Corp'n., 202 F. 2d 642 (C. A. 4, 1953);

Black & Decker Mfg. Co. v. Baltimore Truck Tire Service Corp., 40 F. 2d 910 (C. A. 4, 1930);

Watson v. Heil, 192 F. 2d 982 (C. A. 4, 1951).

Koppers objects to this interrogatory because it is too broad and, in reality, a fishing expedition in an attempt to discover possible acts of infringement outside this judicial district which have no relevancy to the present issues; because it calls for information about installations prior to the issuance of the patent in suit; because it demands information going only to the question of damages to which plaintiff is not entitled unless and until validity and infringement in this district have been established. Koppers argues that the Court, upon objection, should restrict interrogatories to facts that clearly appear to the Court to be relevant in the light of the issues in the case before it, and should weigh obvious burden and expense imposed upon the party interrogated by a nebulous, doubtfully relevant, and apparently speculative interrogatory, against the mere possibility of some undisclosed and remotely possible benefit to the one filing it.

Both plaintiff and Koppers appear to rely upon the case of Martin v. Fairchild Corp., et al., 23 F. Supp. 823 (E. D. N. Y. 1938), as supporting their respective positions with respect to interrogatory No. 1 (and others hereinafter considered). There, non-resident corporations and a resi-

dent defendant corporation were involved and the case was decided under old Section 109 of Title 28 U. S. C., prior to code revision. The Court held that, as to the non-resident defendant corporations, "jurisdiction rests upon their having regular and established places of business within this district and the commission by each of an act of infringement, also within this district". The Court further said: "As to the non-resident defendant corporations over which jurisdiction will fail unless the infringing act be shown, *these interrogatories will be restricted to whatever has been done within the scope of the inquiry, within this district*". (Emphasis supplied.) It is noted that validity of the patent, in addition to infringement, was an issue in that case.

It is well settled that a presumption of validity attaches to the due grant of the patent by the Patent Office. The cases from the Fourth Circuit cited by the plaintiff seem to support the proposition that an additional presumption arises from certain facts, namely, that the invention filled a want arising from a new situation, that it entered into immediate use, that it met with pronounced commercial success; that, in addition, is the presumption arising from the imitation of the patented article by the manufacturer of the alleged infringing device.

However, here the plaintiff is clearly entitled to all pertinent information concerning alleged infringement installations within this district and has already been furnished with certain drawings and other information concerning same. If he desires additional information in that particular, he may obtain it by following proper discovery procedure. The plaintiff says that he has already investigated two other alleged infringing Koppers' installations in other states and it is to be assumed that he can testify relative thereto.

Armed with the presumption of validity of his patent and with other presumptions which may flow from evi-

dence now in his possession or readily available, he appears to be going far afield in interrogatory No. 1. He seeks to obtain information as to all installations, by Koppers, of coke-oven by-product apparatus without this district, for long years prior to the date of plaintiff's patent, without limitation as to the type of saturator here involved. Even if the relevancy of such inquiry were clearly apparent and, according to plaintiff's theory, bore upon the question of "invention", much or all of it might well be merely cumulative and not necessary to a determination of the issues first to be considered. The Court is of the opinion that this interrogatory is too broad in scope the results sought to be obtained are too vague and indefinite and the relevancy is not readily and clearly apparent. Therefore, the objection of Koppers to answering interrogatory No. 1 is sustained.

Plaintiff's interrogatories Nos. 2 and 3 are based upon interrogatory No. 1 and, in view of the Court's ruling, Koppers need not answer these interrogatories.

Interrogatories Nos. 4, 5 and 6 seek information concerning an installation by Koppers of apparatus for the production of ammonium sulphate from coke-oven gas for Pittsburgh Coke & Chemical Company in the State of Pennsylvania, covering a period from January 1, 1945, to January 1, 1950. These interrogatories pertain to one of the installations which plaintiff says he has investigated and, for that and other reasons hereinbefore stated as to interrogatory No. 1, Koppers need not answer them.

Interrogatories Nos. 7, 8 and 9 seek information concerning an installation by Koppers of apparatus for the production of ammonium sulphate from coke-oven gas for Woodward Iron Company in the State of Alabama, covering a period from January 1, 1945, to January 1, 1950. These interrogatories pertain to one of the installations which plaintiff says he has investigated and, for that and other reasons hereinbefore stated as to interrogatory No. 1, Koppers need not answer them.

Opinion Re Interrogatories.

Koppers has already answered that portion of interrogatory No. 10 which pertains to the installation for Wheeling Steel in the Northern District of West Virginia, and Koppers need not answer those portions of this interrogatory pertaining to installations for Pittsburgh Coke & Chemical Company and Woodward Iron Company.

As to plaintiff's interrogatory No. 11, Koppers will be required to answer and its objection to said interrogatory is overruled.

The Court is advised that certain drawings have been furnished by Koppers to the plaintiff relative to the installation of the present apparatus at Wheeling Steel, which drawings appear to satisfy the plaintiff's demands. Unless it is made to appear that these drawings are insufficient for plaintiff's purposes, the Court is of the opinion that that portion of plaintiff's interrogatory No. 12 has been satisfactorily answered. As to the remainder of plaintiff's interrogatory No. 12, Koppers will be required to answer and its objection thereto is overruled.

Koppers' objections to plaintiff's interrogatories Nos. 13 and 14 are overruled and Koppers will be required to answer.

Plaintiff's interrogatory No. 16 is as follows: "Identify any publications by Koppers Company, Inc., or others since January 1, 1945, describing apparatus for sale or installation by Koppers Company, Inc., for the production of ammonium sulphate from coke-oven gas, giving the name and date of publication or other description thereof". Koppers objects to answering this interrogatory because it includes publications by third parties and because it seeks to require an interpretive conclusion as to the contents of such publications. Koppers need not answer that portion of this interrogatory which applies to publications by others. However, Koppers will be required to answer the remainder of the interrogatory and its objections thereto are overruled.

Order Re Interrogatories.

Plaintiff's interrogatory No. 17 is as follows: "Identify any advertisement or brochure prepared or issued since January 1, 1945, by or under the direction of Koppers Company, Inc., describing apparatus for sale or installation by Koppers Company, Inc., for the production of ammonium sulphate from coke-oven gas, giving the date and number or other description thereof". Koppers shall be required to answer this interrogatory and its objections thereto will be overruled.

Attorneys for Koppers are directed to prepare an order consistent with this opinion and, after exhibiting the same to counsel for the plaintiff, present it to the Court for entry.

September 1, 1955.

Order Re Interrogatories.

After due consideration of Plaintiff's Interrogatories and of Koppers' Answers to Plaintiff's Interrogatories and Objections to Plaintiff's Interrogatories, and of the briefs filed relative thereto,

It is ordered that—

Koppers need not answer Interrogatories 1 to 9, incl.

Interrogatory 10 has been sufficiently answered.

Interrogatory 12 has been sufficiently answered as to drawings. Koppers must answer the remainder of this interrogatory.

Koppers has answered Interrogatory 15.

Koppers must answer Interrogatory 16 insofar as it calls for publications by Koppers personnel.

Koppers must answer Interrogatories 11, 13, 14 and 17.

All answers required of Koppers by this order must be served on opposing counsel by October 15, 1955.

Ordered this 23rd day of September, 1955.

/s/ Herbert S. Boreman
United States District Judge.

Opinion on the Merits.

O'BRIEN & O'BRIEN, Central Union Building, Wheeling, West Virginia; PENNIE, EDMONDS, MORTON, BARROWS & TAYLOR, 247 Park Avenue, New York 17; New York; and JOHN E. HUBBELL, 8 West 40th Street, New York 18, New York, for plaintiff.

SCHMIDT, HUGUS & LAAS, Central Union Building, Wheeling, West Virginia, and BROWN, CRITCHLOW, FLICK & PECKHAM, 1706 First National Bank Building, Pittsburgh 22, Pennsylvania, for defendants.

BOREMAN, District Judge:

This is an action for patent infringement, involving two patents owned by the plaintiff, Dr. Carl Otto (hereinafter sometimes referred to as "Otto"), who is a citizen of the State of New York, an engineer, and the head of Otto Construction Company, a New York firm engaged in the design and erection of by-product coke oven plants. Defendant, Koppers Company, Inc., a Delaware corporation (hereinafter sometimes referred to as "Koppers"), has principal offices in Pittsburgh, Pennsylvania, is licensed to do business in West Virginia, and is also engaged in the design and erection of by-product coke oven plants. Defendant, Wheeling Steel Corporation, a Delaware corporation (hereinafter sometimes referred to as "Wheeling"), owns and operates a plant at Follansbee, in the Northern District of West Virginia, where apparatus and processes charged to infringe both Otto patents, have been built and operated.

The original complaint charged infringement of Otto's U. S. Patent No. 2,599,067 which discloses and claims method and apparatus for the production of ammonium sulphate as a by-product from coke oven gas by means of a "spray" type saturator. After answer by Koppers and Wheeling challenging validity of the claims of the patent and denying infringement, Otto served on Koppers a number of interrogatories, to some of which Koppers objected.

his Court ruled on these objections, requiring answers some and sustaining objections to others. Based upon formation thus obtained by Otto through interrogatories, n amended complaint was filed expanding the original omplaint to include an additional ch rge that the de-endants had infringed an earlier Otto U. S. Patent No. ,423,794, which discloses and claims apparatus and process or production of ammonium sulphate as a by-product from oke oven gas by means of a special "flare mouth" type lip pipe saturator. The defendants answered this amended omplaint denying infringement of the additional patent s well, asserting that its claims were also invalid, and pecifying the particular prior art upon which defendants would rely to sustain their charge of invalidity as to both atents.

In considering Kopper's objections to interrogatories prior to the filing of Otto's amended complaint, it became necessary to determine the territorial scope of proof of alleged acts of infringement of Patent No. 2,599,067 and this Court's decision on that question is reported in Otto v. Koppers Company and Wheeling Steel Corporation, 134 Fed. Supp. 886. By the effect of said decision, evidence of infringement as to both patents is limited to structures built and processes used by these defendants in the Northern District of West Virginia.

Explanation of the subject matter of the two Otto patents here involved appears to be necessary. The gas produced from the coke manufacturing process contains ammonia in substantial quantities. For certain reasons, it is desirable, or even necessary, to remove the ammonia from the coke oven gas before the gas can be properly and safely utilized. Over the years, the by-product coke oven industry has faced the problem of removing the ammonia from the gas and at the same time obtaining a by-product disposable to economic advantage.

Opinion on the Merits.

The generally accepted solution of this problem has been to bring the coke oven gas containing the ammonia into contact with a liquor containing sulphuric acid so that a reaction occurs between the ammonia and sulphuric acid, thus producing marketable ammonium sulphate.

For years, most of the by-product ammonium sulphate in this country was produced in saturators in which the gas was bubbled through a bath of liquor containing sulphuric acid. This bath consisted of an acidified saturated solution of ammonium sulphate containing solid crystals of ammonium sulphate, and the effect of the ammonia reacting with the acid was to produce, directly in the bath, more crystalline sulphate. Crystals were periodically withdrawn and acid replaced to enable the process to proceed substantially continuously. Because some solid salt was constantly being deposited on the apparatus itself, it was necessary periodically to "kill" the bath, or to so dilute the bath with water and acid that such deposits would dissolve. The "kill" did not interrupt the removal of ammonia from the gas which continued to flow and the acid-ammonia reaction continued, but under such conditions of dilution that no crystal formation could take place. The pipe through which the gas passed into the liquor in this conventional apparatus was called a "crackerpipe" and usually dipped some twenty-five to thirty-eight inches into the liquor bath. The result was that the gas had to be forced or pumped against a high pressure head in order for it to pass through the crackerpipe against the seal of the liquor bath.

It seems to be undisputed that the early saturators, known as the crackerpipe type, were satisfactory when installed, removed a high percentage of the ammonia from the gas and apparently continued to be satisfactory for many years. The true background history appears to be that soon after 1939, the problem of handling more coke oven gas became acute because the steel industry was put-

tion in more coke ovens. The problem which Dr. Otto sought to solve arose soon after he came to this country in 1939 and he stated this problem as follows:

> "The problem was that it was the trend of the times, by-product plants were getting too small for the gas output * * *. Therefore, it was important to design apparatus which could take care of the conditions to get bigger output through the by-product plants, resulting in remodeling the old by-product plant" (Trans., p. 160).

It was only natural that the producing companies should try to keep the cost of producing ammonium sulphate as low as possible, and it follows that a reduction in the high pressure head would reduce the power cost of pumping the gas through the liquor bath in the saturator.

While both patents in suit relate to ammonia saturators and the incidental production of ammonium sulphate crystals, each patent describes and claims different saturator apparatus and different methods of operation. The respective defenses to the charges of infringement of the two patents involve different prior art, different issues of patentability and different issues of infringement. Therefore, each patent, the claim of infringement and the defense thereto will be separately considered.

U. S. Patent No. 2,423,794

(Otto Flare-Mouth)

[Since there is no appeal from the decision on this patent, the portion of the opinion relating only to it is omitted.]

Opinion on the Merits.

U. S. PATENT No. 2,599,067

(OTTO SPRAY)

The application for this patent was filed on March 15, 1948, and United States Letters Patent No. 2,599,067 were duly and legally issued on June 3, 1952, to plaintiff as the inventor of the invention in production of ammonium sulphate therein described and claimed. The patent as issued contains nineteen claims and plaintiff charges that the defendants infringe basic method claims 1, 2, 4, 7 and 8 and basic apparatus claims 14, 15, 16, 18 and 19. The claims here involved are set forth in footnote 1. Plaintiff's counsel withdrew the charge of infringement of all other claims by letter to the Court dated February 10, 1956, even though the pre-trial conference order set forth the charge of infringement of claims in addition to those appearing in footnote 1. The claim of infringement arises from an installation of coke oven gas, spray type, saturator equipment by defendant Koppers for defendant Wheeling in Brooke County in the Northern District of West Virginia. The installations were made during the year 1952 and continue in use.

This patent pertains to methods and apparatus for removing ammonia from coke oven gas by the use of liquid sprays and the production of ammonium sulphate. Otto testified that he made the invention therein described in May of 1946. Therefore, all public knowledge of the art prior to that date is pertinent to the issue of validity.

The claims in Otto's original application were directed broadly to passing, by means of spray nozzles, mother liquor (saturated acidulated ammonium sulphate liquor) through coke oven gas in an unobstructed scrubbing space, passing coke oven gas through that space, stirring the

1. [These claims are not reproduced here as they appear in the copy of the patent in suit bound herewith at page 369.]

super-saturated mother liquor in a crystallizing pool, removing the large crystals of ammonium sulphate, and respraying the mother liquor, containing entrained crystals, through the scrubbing space. Prior to the filing of this original application for letters patent, Otto filed other applications concerning the same subject matter and bearing the numbers and the respective filing date of each as follows:

Application 659,444 filed April 4, 1946;
Application 668,392 filed May 9, 1946;
Application 674,334 filed June 4, 1946;
Application 720,593 filed January 7, 1947;
Application 771,196 filed August 29, 1947;
Application 3,133 filed January 19, 1948.

The prosecution of the application for this patent in suit continued over a period of approximately four years. Otto's original claims and numerous amended claims were rejected by the Patent Office Examiner and were canceled by Otto. The specification was amended and revised and the claims were rewritten to distinguish from the prior art cited by the Patent Office. The prosecution of the application was interspersed with numerous personal interviews and repeated long arguments.

The inventive concept of producing ammonium sulphate by spraying saturated acidulated sulphate liquor into a gas stream passing through an unobstructed scrubbing space in a saturator by means of spray nozzles, thus causing the falling sprayed liquor to contain solid crystals of ammonium sulphate, was disclosed in Otto application 674,334, filed June 4, 1946.

The full invention described and claimed in patent 2,599,067 was reached as the culmination of the successive inventions disclosed in these earlier Otto co-pending ap-

plications. This patent was granted after examination and consideration of the prior art which is shown in the file history. The rejections of certain claims by the Examiner were reviewed by the Board of Appeals.

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	*Name*	*Date*
441,106	Monsanto	Nov. 18, 1890
1,799,478	Peebles	Apr. 7, 1931
1,932,674	Pyzel	Oct. 31, 1933
2,000,038	Schwalenbach	May 7, 1935
2,035,441	Allen et al.	Mar. 31, 1936
2,045,301	Langer	June 23, 1936
2,288,667	Allen et al.	July 7, 1942
2,375,922	Jeremiassen	May 15, 1945
2,409,790	Otto	Oct. 22, 1946
2,424,205	Otto	July 15, 1947
2,424,207	Otto	July 15, 1947
2,450,095	Seebold	Sept. 28, 1948
2,482,643	Tiddy	Sept. 20, 1949
2,549,848	Otto	Apr. 24, 1951

OTHER REFERENCES

Fessenden, The Register of Arts, published in 1808 (page 87), C. & A. Conrad & Co., Philadelphia.

Defendants, in their amended answer, deny validity of the invention claimed, alleging that every material and substantial part thereof was disclosed in publications and patents prior to Otto's alleged invention, and was patented or described in printed publications or in public use in this country more than one year prior to the application for this patent, as shown by the following:

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"Patents"

Brunck	U. S. 824,092	Patented	June	26, 1906
Doherty	U. S. 997,908	"	July	11, 1911
Bosch	U. S. 1,029,528	"	June	11, 1912
Eneas	U. S. 1,101,264	"	June	26, 1906
Becker	U. S. 1,307,534	"	June	24, 1919
Becker	U. S. 1,375,483	"	April	19, 1921
Becker	U. S. 1,654,159	"	Dec.	27, 1927
Becker	U. S. 1,747,616	"	Feb.	18, 1919
Berkhuijsen	U. S. 1,985,010	"	Dec.	18, 1934
Berkhuijsen	Swiss 172,661	"	Oct.	31, 1934
Tiddy	U. S. 1,997,757	"	April	16, 1935
Jeremiassen	U. S. 2,375,922	"	May	15, 1945

"Publications"

Hutte Handbook (German)	Published in 1927 by Verlag Von Wilhelm Ernst and Sohn, Berlin

"Prior Inventor"

Charles E. Underwood	Bethlehem Steel Co. Bethlehem, Pennsylvania

"Prior Public Use"

Bethlehem Steel Co.	Bethlehem, Pennsylvania

Koppers and Wheeling contend: That the Patent Office was misled by Otto's incorrect argument concerning "Whirling sprays"; that there was nothing novel or inventive with Otto in 1946 in the use of liquid sprays; that Koppers had used sprays in saturators before 1946; that there was no invention in scrubbing coke oven gas with sprays in an unobstructed space; and that there was noth-

ing novel or inventive in spraying mother liquor that contained ammonium sulphate crystals. It is the position of these defendants that this invention claimed by Otto was merely a "non-inventive" engineering evolution, using methods and apparatus known in the prior art.

It is a well established rule of law that the grant of the patent is prima facie evidence of its validity. 35 U. S. C. A. 282; Diamond v. Consolidated, 220 U. S. 428, 434 (1911); Baker-Cammack Hosiery Mills v. Davis Co., 181 F. 2d 550, 564 (4 Cir. 1950), certiorari denied 340 U. S. 824; Chesapeake & Ohio Ry. Co. v. Kaltenbach, 95 F. 2d 801 (4 Cir. 1938); McKee v. Graton & Knight Co., 87 F. 2d 262 (4 Cir. 1937).

The presumption of validity is strengthened where the patent was granted by the Patent Office after a consideration of and extensive administrative proceedings concerning, the same prior art as that relied upon by the defendants, or similar thereto. Hall v. Montgomery Ward & Co., 57 F. Supp. 430 (N. D. W. Va. 1944); Modern Products Supply Co. v. Drachenberg, 152 F. 2d 203 (6 Cir. 1945), certiorari denied 327 U. S. 806; Hildreth v. Mastoras, 257 U. S. 27 (1921); Reynolds v. Whitin Mach. Works, 167 F. 2d 78, 83 (4 Cir. 1948), certiorari denied 334 U. S. 844; Hoeltke v. C. M. Kemp Mfg. Co., 80 F. 2d 912, 919 (4 Cir. 1936), certiorari denied 298 U. S. 673.

The judgment of the Patent Office officials in granting the patent in view of the prior art is entitled to great weight and is to be overcome only by clear proof that they were mistaken and that there is a lack of patentable novelty. Hall v. Montgomery Ward & Co., supra; Gulf Smokeless Coal Co. v. Sutton, Steele & Steele, 35 F. 2d 433 (4 Cir. 1929), certiorari denied 280 U. S. 609; Patterson Ballagh Corp. v. Moss, 201 F. 2d 403 (9 Cir. 1953); Lever Bros. Co. v. Procter & Gamble Mfg. Co., 139 F. 2d 633, 640 (4 Cir. 1943).

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However, it is well established also that, even though the patent is presumed to be valid, the presumption may be overcome by new references to prior art not considered by the Patent Office. Todd v. Sears Roebuck & Co., 216 F. 2d 594 (4 Cir. 1954); Gomez v. Granat, 177 F. 2d 266 (9 Cir. 1949); Friend v. Walsh, 141 F. 2d 180 (2 Cir. 1944).

From the record in this case, from the file history of proceedings before the Patent Office Examiner and the Patent Office Board of Appeals, from written briefs and arguments appearing in the file wrapper identified as Plaintiff's Exhibit 1, and from the briefs and arguments submitted here, this Court concludes that Otto's concept of his invention can be simply described as follows: Passing through and from a spray nozzle or spray nozzles, under pressure, an acidified saturated solution of ammonium sulphate (containing entrained ammonium sulphate crystals) in the form of a finely divided spray through coke oven gas containing ammonia while the gas is being pumped or forced through an unobstructed space in what is commonly known to the art as a saturator.

On page 34 of plaintiff's main brief appears this heading: "The Disclosure of Patent 2,599,067". Then near the bottom of the same page and continuing on page 35 appears the following:

> "The particular spray disclosed in the drawings and specification of Patent 2,599,067 is a 'whirling spray', defined as the type of spray produced by a Spraco nozzle such as shown in the Eneas Patent 1,101,264 (PX2; PX26A and 26B).
>
> "Unobstructed space is disclosed as a space in which there is no obstacle to impede the gas flow from its inlet to its outlet . . .
>
> "The sprayed liquor with which Dr. Otto is concerned is an acidified saturated solution of ammonium sulphate containing entrained crystals."

"It is important to remember these three distinctions in examining the prior arts. It will then be clear that no patent nor publication nor prior use discloses a saturator embodying *these three features.*" (Emphasis supplied.)

It appears clearly that Otto did not invent the type and kind of spray nozzles used in his patented method of spraying the ammonium sulphate solution through the coke oven gas. He purchased these on the open market from the manufacturer and these spray nozzles themselves are described in U. S. Eneas Patent 1,101,264, and further described in pages from a catalogue of the manufacturer, Spray Engineering Co., of Somerville, Massachusetts, submitted to the Patent Office Examiner at an oral interview on August 1, 1950, and shown in Plaintiff's Exhibit No. 2. Excerpts from Otto's testimony on cross-examination, taken from pages 199 and 200 of the transcript, are quoted as follows:

"Q. • • • What do you mean and what do you understand by the word 'spray' in your patent? A. A subdividing of liquor, or a subdividing of a stream of liquor in small particles."

• • • • •

"Q. You didn't invent anything yourself in regard to sprays or spray nozzles, so far as your patent on the spray type saturator is concerned, did you? A. No. In connection with the spray type saturator, no, I did not invent a new design of a spray.

"Q. As a matter of fact, the sprays that you used and referred to in your patent, and that you use in your saturators, had been on the open market and on sale long before your alleged date of invention in the spray patent, is that right. A. That's right.

"Q. And you simply bought them up yourself and put them in your saturator, so to speak? A. That's right.

"Q. And you can buy them—they will give you most any kind of spray you want—can't you—coarse sprays, fine sprays? A. That's right.

"Q. Streams, mists, atomizers or what not? A. That's right.

"Q. And that was all well long before anything you claim in either patent? A. That's right."

Further excerpts from Otto's testimony on cross-examination, beginning on page 209 and continuing through page 212, follow:

"Q. Now, Doctor, the essential feature of the Otto spray saturator, as you understand it, is the spraying of sulphuric acid through an unobstructed space through which coke oven gas passes, isn't it? A. This spraying is not of sulphuric acid. The spraying of an acidified ammonium sulphate solution through an unobstructed space.

"Q. Well as I understand it, you have modified my statement by defining the liquid that is sprayed to be an acidified solution of sulphuric acid containing ammonium sulphate? A. That is right.

* * * * *

"Q. Now the liquor you are talking about is mother liquor that has previously passed through the saturator and has been drawn off at some place and then is recirculated. Isn't that right? A. That is right.

"Q. So that the essential feature then is spraying liquor containing sulphuric acid and ammonium sulphate crystals through coke oven gas. Is that right? A. Not mainly sulphuric acid, but mainly mother

liquor containing crystals, this liquor being acidified with sulphuric acid. Sulphuric acid is a small proportion of this liquor.

"Q. Well, the liquor that is circulated in your spray type of saturator is simply a part of the mother liquor that has already been used, is it? A. That's right.

"Q. Plus the addition of some sulphuric acid? A. That is right.

"Q. To make up for what has been lost by ammonium sulphate crystals that have been removed? A. That is right.

* * * * *

"Q. The same kind of mother liquor also resulted from the use of the crackerpipe saturator? A. It is the same type of mother liquor as in a crackerpipe saturator.

"Q. So what I am coming at and getting to, with some circumvention as part of my mistake, sir, is that the real crux of the invention which you are asserting is the spraying of a certain type of liquor through coke oven gas containing ammonia for the purpose of removing the ammonia. Isn't that right? A. That is right.

"Q. Now the question I was leading up to, the auxiliary matters—apparatus such as tar catchers, acid catchers, crystal separators, crystal graders, and so forth, is conventional apparatus not peculiar to spray type of saturators at all. Isn't that right? A. That's right.

"Q. And such apparatus was old and well known in the art and can be of any conventional type, as I understand it. A. Right.

"Q. And can be positioned at any particular place in the installation that is convenient and engineeringly satisfactory. Is that right? A. Right.

"Q. And not dependent upon any invention on your part as to where they are placed? A. These additional parts are not part of my construction.

"Q. So that what you really want to do by asserting this 067 patent is to prevent everybody else from spraying mother liquor through coke oven gas in an unobstructed space. Isn't that right? A. That is in so far right as we do not want anybody to do this without cooperation with us."

In Otto's deposition filed July 1, 1955, and identified as paper 22 in the court file, pages 40 through 43, the following questions and answers appear:

"Q. Now, aside from washing, what did you do to avoid the difficulties which you say had confronted you in using this spray-type of getting contact of the liquor—we will call it—with the coke oven gas? I think you said in the beginning that you knew sprays were old. You knew sprays were old even for coke oven gas. But you were confronted with certain difficulties. A. That's right.

"Q. I asked you how you knew of these difficulties, and as far as I recall, the only one you referred to was washing the walls. What other difficulties did you meet, and how did you solve them? A. Difficulties, to have a spray which was non-clogging, and I picked the spray which was known in the art, you see, this special Eneas construction, and where I expected to have no difficulty with clogging up. They call it a non-clogging spray.

"Q. As I understand it, you went out and picked out, in the art, a commercial spray which would give you what you wanted—a spray nozzle? A. One which I hoped to give me what I hoped to get. You see, it could just as well happen that this spray, for example, would have, maybe, clogged up.

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"Q. Did it give you what you hoped to get? A. Yes, it gave me, because I don't remember that that spray—that not a single spray in our saturator has ever blocked up.

"Q. And the spray that you bought on the open market gave you the results you wanted, without any modification by you—is that right? A. That's right.

"Q. Your patent speaks several times about spraying liquor through an unobstructed space. Just what do you understand or what do you mean to say by those words? A. That means, empty. As I understand, it is empty space. That means, no grids, or other filling, you understand, or wooden grids.

"Q. Well— A. (Interposing) That is well known in the art, where you can put in, for instance, tiles.

* * * * *

"Q. I think, Dr. Otto, that you are taking my question to mean more than I did, and making it more complex than necessary. I want to know if what you meant by an unobstructed space was simply that your spray went into a gas chamber so that the spray could pass through the chamber without having to go to other surfaces, other than the walls of the tank, until it reached the liquor at the bottom of the tank. Is that what you mean? A. That's right."

At page 247 of the transcript, Otto testified to the same effect concerning "unobstructed space".

At page 60 of Otto's deposition, the following questions and answers appear:

"Q. I think you answered my question with a sentence four or five sentences back, when you said that once an engineer was told that he could remove ammonia from coke oven gas by spraying a liquid through the gas, the making of the apparatus would be just an

engineering matter that any good engineer could do—is that right? A. Could design the apparatus to do it, you see. But nobody decided on that—that it was possible to do that.

"Q. Well, doesn't that mean, Doctor, that your position is that the essence of what you regard as your invention is spraying a suitable liquor through coke oven gas to remove— A. (Interposing) That's right.

"Q. —to remove ammonia? A. To spray saturated liquor containing crystals through nozzles in a fineness to give enough contact, through coke oven gas.

"Q. Well, I have to go back: the answer is yes. Now, the question of fineness (of spray), and so forth, is a matter that can be worked out by anybody by experiment? A. Yes."

At pages 78 and 79 of the same deposition appear the following questions and answers:

"Q. Well, is it fair to say, Dr. Otto, that the characterizing feature of any of the saturators you have built, is the use of the spray through an unobstructed chamber, through which the gas passes? A. Yes.

"Q. They have that in common? A. Oh, yes, they have that all in common.

"Q. Now, leaving that feature out, as I understand it, the other mechanical elements can be arranged in a great variety of forms and positional relations to each other—is that fair to say? A. Yes, that's right, but all have this feature what you have just mentioned.

"Q. And if you took that feature out, it wouldn't have much except just an engineering building proposition, would it? A. Well, I don't know if other features, perhaps, can also get some patents. It depends on the Patent Office, you see—other additionals which come up.

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"Q. What I am getting at is, your idea of what is the essential idea of all your apparatuses that you have built. A. This is a fundamental, main feature."

At page 86 of the same deposition, the following appears:

"Q. What I am asking you is—and it is the only point that I had in mind—there is no virtue in one spray as distinguished from two sprays or half a dozen sprays, or whether they are arranged around in a circle, or whether there is one in the middle that covers the whole chamber? A. Mr. Brown, I would only say this: if you make too many sprays, then the sprays are so small, then you come to a point where the sprays will block up. I couldn't tell you where that point it. Then you find out, according, where you would have difficulties.

"Q. So, you can make any arrangement of sprays, with a wide variety, a wide range of variations, and still get satisfactory results? A. That's right."

On cross-examination, Otto testified as follows:

"Q. I want to know if you have done something and taught something in your patent that makes the use of the spraying of a solution of sulphuric acid through coke oven gas more effective, more efficient, more valuable or different in any way from prior spraying of sulphuric acid solutions through coke oven gas, and we will prove that that has been done, I think. A. In this general way I could not answer that question" (Trans., page 227).

* * * * *

"Q. Doctor, is it fair to say that the crux and essence of what you regard as the invention described and claimed in the spray patent in suit is the use of

sprays exclusively to take ammonia out of coke oven gas? A. You talk about the crux and essence?

"Mr. Brown: Please read the question.

"A. Our invention, or our process, is described clearly in the claims of our patent and that is not as broad as I think you just expressed yourself. It is the spraying of acidified saturated liquor. That means liquor of dissolved ammonium sulphate containing crystals in an unobstructed space.

"Q. That is the crux and essence of your invention as you have just stated it? A. That's right.

"Q. And that invention, as you understood, would be infringed regardless of the apparatus that was used to practice it, provided the process met the definition you have given. Is that right? A. That is right. That is the process claimed. I see here in our claim it is also inferred that this liquor gets supersaturated. It is also mentioned in there" (Trans., pages 267 and 268).

Claim 1 of this patent includes as an element "said contacting being affected by spraying by whirling motion and under such pressure so as to disperse said solution in fine spray". Otto was interrogated at some length concerning the spraying by whirling motion and his answers were somewhat evasive. He seemed unwilling to admit that there is no whirling motion to the spray droplets of liquor after they leave the spray nozzle. At the bottom of page 213 and at the top of page 214 of the transcript appear the following question and answer:

"Q. The individual particle, when it leaves the nozzle, would travel in a straight line except as gravity may tend to draw it towards the earth, or currents of gas might move it sideways, but otherwise it moves

in a straight line. Is that right? A. I could not decide that. I think these special companies, if you are interested in that, they will tell you what these drops do. I know that the drops by this power of energy are separate and in small particles, which is the object of my construction."

At page 216 of the transcript, after further interrogation concerning the line followed by individual droplets, in answer to this question, "Disregard gravity and disregard currents of air, it would go in a straight line"? Otto replied, "That's right, it would".

Otto's final conclusion concerning "spraying by whirling motion" was summarized at page 296 of the transcript on re-cross examination as follows:

"Q. Doctor, so that there may not be any mistake, my understanding is you testified yesterday and again today that any whirling there was of the liquid occurred inside of the nozzle? A. That's right.

"Q. All before it passed out of the nozzle? A. That's right.

"Q. So that there wasn't any whirling of the particles in the sense of projecting them in a spiral fashion after they left the nozzle? A. No."

Claims 2 and 4 of the patent, by reference to Claim 1, contain the same element—"spraying by whirling motion". It should be explained that Claim 1 in the patent is the same as Claim 61 presented to the Patent Office. Claim 2 is the same as Claim 62, and Claim 4 is the same as Claim 64.

The Court is convinced that the Examiner understood from Otto's presentation and arguments that the inventive feature of these claims, Nos. 1, 2 and 4 and even others, was based on the novelty and value of that whirling spray element.

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An amended application was filed by Otto with the Patent Office, the oath thereto being dated September 25, 1951, which refers to interviews between the applicant and Examiner on August 1 and August 20, 1951. Certain portions of the application are quoted as follows:

> "At the interview which the applicant had with the Examiner on August 1, 1951, the Examiner expressed his willingness to allow the Claims 61, 62, 63 and 64 of the present amendment, in addition to the Claims 65-68 and 71-73 which the Examiner had previously found allowable.
>
> "In accordance with the requirements of Rule 133(b), the reasons and explanations presented by the applicant at the above mentioned interviews as warranting favorable action on applicant's claims, are set forth in subsequent portions of these 'remarks'.
>
> "At the August 1, 1951, interview, it was pointed out by the applicant that his practice of dispersing the liquor passed into the scrubbing space by a whirling motion as specified in Claim 61, is readily effected by the use of spray nozzles of standard forms, and in particular by the spray nozzles of the well known type disclosed in the Eneas patent 1,101,264.
>
> "The foregoing references to the statements made by the applicant at the August 1, 1951, and August 20, 1951, interviews are based on the applicant's present recollection refreshed, as the Examiner will understand, by reference to draft amendments which the applicant had with him at the interviews and which the applicant had helped to prepare."

In his brief filed by Otto's attorney before the Board of Appeals, it is stated:

"In the commercial use of his invention, the applicant uses spray nozzles of a well known type formed to discharge spray liquid with a whirling motion. While the applicant prefers to use such nozzles, there are other well known forms of spray nozzles which do not discharge spray with a whirling motion."

Prior to the filing of this brief, the Examiner said:

"Claim 61 defining the particularly whirling motion of the finely divided spray droplets, stated to give an improved contacting between the crystals containing liquor and the gas being treated and thereby an improved yield, is considered to be allowable as are the dependent claims 62-64" (File wrapper, paper No. 20, October 11, 1951).

In the brief of the Examiner filed with the Board of Appeals, it is stated:

"Invention has been conceded in the process Claims 61-64 and 68 which define the whirling motion of the spray droplets as stated to give improved yield as expressed, for example, Claim 68 which recites the spraying at three separate levels with a different type spray at the several levels giving the improved contacting as stated."

The cited Eneas patent, describing the swirling nozzle spray invention, makes it clear that divided liquid streams are given a swirling motion before uniting with a non-swirling stream in a mixing chamber within the nozzle. This causes the blended mass of liquid to issue in a "spray of substantially uniform homogeneity". The description of the Eneas nozzle in the catalogue submitted to the Examiner at the oral interview is in part as follows:

"*The major portion of the liquid* passes through the outer vanes of the stationary turbine center and

> *is given a rapid swirling motion,* The remainder of the liquid passes straight through the center-jet and strikes the outer rotating mass just below the orifice in the mixing chamber, causing the liquid to issue from the nozzle in a full conical formation of equal density across its section." (Emphasis supplied.)

There is no claim in the Eneas patent, or in the descriptive material furnished by the manufacturer, that the sprayed droplets have a whirling motion after they leave the nozzle.

At the trial it was shown that the liquid droplets have no whirling motion at the time they leave the spray nozzle and as they enter the gas in the unobstructed scrubbing space. However, Claims 61, 62 and 64, as allowed (Claims 1, 2 and 4 of Otto's patent), were rewritten in amended form, were presented to the Examiner by Otto's attorney at the time of the August 1951 interview and there, for the first time, the *contacting of the gas and liquor is described as effected by spraying the liquor by whirling motion.*

Plaintiff, in his trial brief, states:

> "Koppers did not realize the importance of having a 'whirling' spray such as produced by Spraco nozzles * * *."

In his main brief, he states:

> "The particular spray disclosed in the drawings and specification of Patent 2,599,067 is a 'whirling spray', defined as the type of spray produced by a Spraco nozzle such as shown in the Eneas patent * * *."

In plaintiff's reply brief, he refers to the "myth of the whirling spray" and terms it a "straw man" set up by

the defendants in order to knock it down. He charges that the defendants imply that it must be understood as meaning that the sprayed droplets would move in a spiral path after leaving the nozzle. After further written argument, he states:

> "in the face of this, it is difficult for plaintiff to understand how defendants could hope to succeed in impressing the Court with their *whirling spray* 'straw man' * * *."

From this Court's understanding of the meaning of the words "spraying by whirling motion" and the view obviously presented by Otto's patent attorney to, and accepted by, the Examiner, it would seem that the "straw man" is, rather, a substantial giant. It is clear from the file history that the reason which prompted the inclusion of the "whirling spray" element of Claims 1, 2 and 4 was based on the theory that the *contacting* and the *resultant better yield* were accomplished by *spraying the mother liquor by whirling motion.* There could be no *contacting* of liquor and coke oven gas in the "Unobstructed space" until *after* the liquor left the nozzle in the form of spray. The only whirling motion of the liquid was within the nozzle itself, which was designed to break the liquid into drops of uniform size.

In considering further the questions here presented, the Court will assume that the reference in Claims 1, 2 and 4 to "spraying by whirling motion" was adopted for the sole and only purpose of identifying the particular type of spray nozzle used by Otto, and that he and the Patent Office were in full understanding and agreement as to such purpose, as he now argues. There remain three major assertions of invention—(1) spraying the mother liquor, to which sulphuric acid has been added, *in the form of finely divided spray,* (2) over substantially the whole cross-

sectional area of the *unobstructed space* through which coke oven gas is passing, (3) said mother liquor containing *entrained crystals* of ammonium sulphate.

In plaintiff's reply brief under the heading, "What The 'Spray Saturator' in This Case Really Is", the following appears on page 17:

> "A spray saturator is a specific piece of apparatus accomplishing not only the function of scrubbing ammonia out of coke oven gas, but also the function of simultaneously producing by chemical reaction a useful by-product, ammonium sulphate, in solid crystal form. *The specific combination which characterizes the apparatus and method of Dr. Otto's invention is found nowhere in the prior art although all of the elements which go to make it up were old and readily available to workers in the art.*" (Emphasis supplied.)

Defendants have cited certain patents and other prior art, as hereinbefore shown, which were not considered by the Patent Office. It will not be possible here to discuss all of them in detail but brief reference will be made to some which appear to bear upon the state of the art prior to May 1946, the claimed date of Otto's invention.

Doherty U. S. Patent No. 997,908, issued in 1911, describes a process of obtaining ammonia from coal. A copy of this patent is found in Defendants' Exhibit No. 18, and a chart showing the Doherty structure is identified as Defendants' Exhibit No. 19. Gas produced by heating coal is passed through absorption chambers, and at the top of each chamber is located "a plurality of spraying caps" having perforations in their faces. The streams of liquid discharged from these perforations intersect and impinge upon each other to produce "a very fine spray", which is discharged into the gas passing through an open unobstructed chamber. The spray is an "ammonia absorbing

liquid" which "will usually be a dilute solution of sulphuric acid, the ammonia absorbed being fixed as ammonium sulphate". Claim 4 of the Doherty patent calls for recovering ammonia from coal gas by subjecting the gas to contact with a (sprayed) liquid capable of absorbing the ammonia. Fresh ammonia-absorbing liquor is "sprayed" into absorption towers at a rate at which saturated liquor is withdrawn from the towers. At page 3, column 1, beginning at line 64 of the patent, Doherty says:

> "It is apparent that my method of absorbing the ammonia insures the most intimate contact possible between the gas and the liquid, the absorption of the ammonia being rapid and complete. I thus reduce the capacity necessary in the absorbing apparatus to a minimum."

Thus, this early patent meets Otto's basic idea of removing ammonia from coke oven gas by passing mother liquor in the form of "a very fine spray" through the gas in an unobstructed scrubbing space, collecting the saturated liquor for treatment in crystallizing equipment, and respraying the mother liquor, in a continuous process. Figure 4 of the drawings indicates that the spray chamber is clearly unobstructed.

Otto states in his main brief, at page 35:

> "Defendants cite Doherty as a disclosure of the use of sprays to remove ammonia from coke oven gas. It does show this, but is none the less irrelevant here because (1) it is only another example of the use of a spray tower in the *un*saturated solution method of ammonium sulphate production, (2) it does not go beyond many such examples considered and discarded by the Examiner, and (3) it differs in important specific respects from the reqaisites of an Otto spray saturator."

Opinion on the Merits.

Bosch U. S. Patent No. 1,028,528 issued in 1912, a copy of which is shown in Defendants' Exhibit No. 18, describes a process of removing oxides of nitrogen from a gas by spraying a liquid, containing solid particles in suspension, through the gas. This is done by "introducing a suspension of an absorbent into the gas mixture in a state of fine division, for instance as a spray, or mist". This Bosch patent makes clear that the use of spray nozzles was old for producing such gas-scrubbing methods. While coke oven gas is not mentioned, it is obvious that the method is similar to that here involved in Otto's patent and that the problems and practices are analogous. The plaintiff says that there is no disclosure in the Bosch patent that the liquid so sprayed is saturated or tends to clog or encrust. He says that it shows spraying a slurry in an unobstructed space to remove nitrogen oxides therefrom; that it adds nothing to Doherty, supra, and the only apparent reason the patent was cited by defendants is as a disclosure of a spray tower.

Becker U. S. Patent No. 1,307,534, issued in 1919, a copy of which is shown in Defendants' Exhibit No. 18, shows diagrammatically a complete by-product coke oven and auxiliary apparatus for continuously treating coke oven gas to remove ammonia and other elements. Three treatment towers are shown each having inlet openings at the bottom and outlet openings at the top for a continuous passage of coke oven gas. In the top of tower 8 piping is shown, apparently used for spraying mother liquor through the gas to reduce the ammonia to ammonium sulphate. Fresh acid is continuously added to compensate for that removed from the system as ammonium sulphate crystals. The mother liquor sprayed into the top of the ammonia absorber tank is pumped from near the bottom of the crystallizing chamber. Consequently, it inevitably contains entrained crystals, just as called for in the Otto patent in suit, as all of the witnesses, including Otto, were

of the opinion that the mother liquor obtained from the crystallizing chamber contains entrained crystals of ammonium sulphate. In Otto's deposition filed July 1, 1955, these questions and answers appear:

> "Q. Don't you know of any patent or publication, prior to 1946, which showed and described apparatus in which coke oven gas was passed through a chamber at the top of which sprays were located, and the sprays were supplied from liquor which was drawn from the bottom of that chamber? A. I only know what you have mentioned to us—this patent of Becker that you refer to.
>
> "Q. I didn't mention a patent of Becker. I am quite sure I didn't. I am sure you did. You brought it up.
>
> • • • • •
>
> "Q. Now, understand, I didn't ask you about the Becker patent but I thought you said that that was—that my question was, in effect, a statement of what Becker shows. Is that what you meant to say? A. Yes, that is what I think.
>
> "Q. That is what Becker shows? Becker shows what I asked you about—is that right? A. That's what I think there."

The deposition, at page 92, further shows that Otto was examining a patent at the time he was so testifying and it was identified as No. 1,307,534. Plaintiff argues that this patent shows ammonia being absorbed out of coke oven gas in a conventional acid washer tower and that there is a clear disclosure that unsaturated sprays were intended and, hence, no crystals would have existed in the spray of the acid washer tower.

The German Hütte handbook for the Practical Chemist, published in 1927, illustrates and describes a "spray scrubber without obstructions" for "scrubbing" gas by passing

it into the bottom of an open tank and out at the top thereof while spraying scrubbing liquid through "spray nozzles" located at the top of the tank. This handbook states that the process and apparatus shown are "satisfactory * * * for carrying out rapidly-proceeding reactions and absorptions". Defendants urge that this reference is clear on its face as to the teaching of a gas scrubbing spray process; that "scrubbing" is a generic term that refers to removing some element from the gas treated; that the Otto spray patent in suit refers to his patented process as a "scrubbing" operation. Plaintiff objects to the reference on the ground that there is no disclosure in the particular section of the handbook cited by the defendants dealing specifically with the production of ammonium sulphate, but the section deals with the general subject of spray scrubbers and absorbers. Plaintiff urges that the most significant thing about Hütte is that he shows open spray towers to be good (although packed towers he says are better) as general chemical engineering tools, but when it comes to ammonium sulphate, he recommends the familiar crackerpipe.

Jeremiassen U. S. Patent No. 2,375,922, issued in 1945, was considered by the Patent Office and was repeatedly cited by the Examiner as meeting Otto's basic claims. It describes a method of treating gas containing ammonia with a liquid containing sulphuric acid whereby sulphate of ammonia is formed. This patent, as does the Otto patent in suit, refers to known methods of removing ammonia, specifically the crackerpipe and the packed tower types of saturators. Jeremiassen treats ammonia-containing gas with sulphuric acid in the form of "liquid streams or jets which, to a greater or less degree, will be disrupted so as to form drops". Fresh sulphuric acid is continuously added to replace that taken out as ammonium sulphate crystals. Jeremiassen states that the liquor

that has passed through the gas is recirculated through the gas. In both of the apparatuses shown in the patent drawings, a part of the "solution" that is passed through the gas is pumped directly from the crystallizing chamber, in which ammonium sulphate crystals are formed. All of the witnesses who testified in this case appeared to be in agreement that the mother liquor bath contains small crystals of ammonium sulphate in addition to the larger crystals which are formed and which settle to the bottom. It is this mother liquor which is recirculated and passed through the gas in the Jeremiassen process. This Court is led to the conclusion that this mother liquor contained entrained ammonium sulphate crystals. Otto's testimony concerning crystals in mother liquor would lead to that conclusion and witnesses Helm and Lowry testified that entrained crystals would be present in Jeremiassen's liquid showers. Plaintiff's expert witness, Professor Radasch, testifying in connection with the Tiddy patent hereinafter mentioned, apparently agreed that crystals exist at the top of a mother liquor crystallizing pool. Jeremiassen teaches that he depends entirely on a finely divided sulphuric acid and ammonium sulphate liquor passed through an unobstructed scrubbing space to react with ammonia and to produce ammonium sulphate. He refers to the liquid entering the unobstructed scrubbing space as a shower of streamlets or jets and creating a veil of liquid through which the gas must pass. He describes his process as producing the desired "very large contacting surface between the liquid and the gas", resulting in the production of ammonium sulphate, the larger crystals of ammonium sulphate settling to the bottom of the crystallizing chamber where they are removed. It is true that Jeremiassen intends to avoid the use of sprays in his process, but plaintiff's expert witness Radasch admitted that whether Jeremiassen's shower of

sulphuric acid was called "sprays" or not, it would be effective to remove the ammonia from coke oven gas. Plaintiff argues that there are at least two primary differences between Jeremiassen's patented process and that of Otto. He says (1) that Jeremiassen does not disclose a process in which crystalline ammonium sulphate is formed in the reaction zone, intending that the contact zone will be crystal free by keeping the circulating fluid within the limits of metastable supersaturation, and (2) he does not disclose that the liquid is dispersed in his reaction zone in a spray.

The record in this case contains much testimony from the expert witnesses who labored with their explanations and understandings of "metastable state" and "limits of metastable supersaturation". The experts certainly were not in agreement as to the exact meaning of these terms, nor were they in agreement as to the effect of the processes.

Of course, Jeremiassen did not use spray nozzles of any kind, certainly not "whirling sprays" as does Otto, but he surely passed the gas through an unobstructed space and passed through this gas a flow of liquid in the form of "streams or jets which, to a greater or less degree, will be disrupted so as to form drops", thus creating a "veil" of liquid through which the gas must pass. Let us assume that no formation of crystals of ammonium sulphate actually occurred in the unobstructed space or the reaction zone, but the fact remains that the ammonia was scrubbed out of the gas by the fluid even though it was "within the limits of metastable supersaturation" and that the ammonium sulphate crystals later resulted from that process.

I turn now to the consideration of Tiddy U. S. Patent No. 1,997,757, issued in April, 1935 (Defendants' Exhibit No. 37), which was cited by the defendants against Otto. This Tiddy patent describes a saturator for pro-

ducing ammonium sulphate by reaction of sulphuric acid with ammonia. He states, as an object, removal of ammonia completely from gas containing it "by contacting such gases with a spray of acid". Tiddy produced his spray of "mother liquor" by rotating a cylinder, the lower portion of which dips in the surface of the liquor bath. He points out that the reaction of ammonia and sulphuric acid takes place at the surface of the liquid which becomes quickly saturated with ammonium sulphate. The rapid rotation of the cylinder, dipping into the liquor bath, throws out a tangentially directed spray, which is shown as completely filling the unobstructed scrubbing space through which the ammonia-carrying gas is passed. The result stated is the complete removal of ammonia. This patent states as an object of the apparatus and method described:

> "To provide a process for the production of ammonium salts from an ammonia-containing gas and a fine spray of a solution containing an acid."

He obviously had in mind the use of sprays of mother liquor for removing ammonia by contact with sulphuric acid solution to give maximum surface contact of gas and liquid and thus avoid "the establishment of back pressure on the stream of gas."

The patent further states:

> "The sprayed liquor containing ammonium sulphate, both dissolved and crystalline, drips down the side of the spray chamber and is returned to the main body of liquid in the reservoir, which liquid thus becomes slowly saturated with ammonium sulphate."

This would seem to be the basic process which plaintiff seeks to cover in his spray patent because, in Otto's

patent, the mother liquor sprayed through the gas drops into a reservoir where the crystals grow in the supersaturated mother liquor, from which the large crystals are separated from the mother liquor. The processes of Tiddy and Otto would appear to be practically the same except that Tiddy creates his spray from the surface of the mother liquor bath, while Otto brings the mother liquor solution into the top of the unobstructed space and sprays the liquor through the gas by means of spray nozzles.

Professor Radasch had been engaged by Otto to make a study of the prior art and to testify as an expert witness at the trial on behalf of Otto. In his direct testimony Radasch made no reference to this Tiddy patent No. 1,997,757 as bearing upon the prior art, but on cross-examination the witness honestly admitted that he had examined the patent and testified as to Tiddy's teaching. The materiality of the testimony of Radasch will be apparent from questions and answers appearing in the transcript at pages 687-91:

> "Q. Did you mean to tell the Court that in your research and study leading up to what you called the evolution of ammonium saturators, that you had found no reference in the published art or patented article of a sulphuric acid solution being passed in finely divided form through ammonia gas for the purpose of removing ammonia prior to the Otto spray patent in suit? A. I found such a reference, yes.
>
> • • • • •
>
> "Q. Well, would you mind telling me what it is now? A. No, sir. It is the Tiddy patent 1,997,757 filed in August, 1931, and granted in 1935.
>
> "Q. What does that patent teach you as a chemical engineer and what, in your opinion, does it show as

to projecting sprayed sulphuric acid solution into a space containing coke oven gas or other gas to be treated by the sprayed liquid? A. Well, it taught me a lot, because I knew something about that development and I knew that Mr. Tiddy had in mind the fact that a spray from a spray nozzle to do this job might not work on the basis of some other experience that he had had and that he devised a particular kind of spray equipment to do this job, which I distinguish from a spray tower type of equipment.

"Q. Well, so far as the interaction of the sprayed sulphuric acid solution into coke oven gas, the fine particles thrown up by the rollers in the Tiddy patent would interact with the coke oven gas and remove the ammonia in the form of ammonium sulphate, would they not? A. That's correct.

"Q. So that there, as you understand it, before the date claimed for his invention of the spray patent by Dr. Otto, which, for your information, I will tell you on the record was in May or later of 1946, so that this Tiddy patent, as you interpret it, showed that a prior inventor had proposed to remove ammonia from coke oven gas by spraying, or call it what you will, projecting fine particles of sulphuric acid solution through the gas? A. Mr. Tiddy very definitely did propose that.

• • • • •

"Q. Professor, this Tiddy patent describes spraying the sulphuric acid through the coke oven gas. You have agreed with that? A. Yes, sir.

"Q. Will you also agree that this sprayed material was picked up out of the mother liquor bath which contained ammonium sulphate crystals? A. We haven't agreed to it yet. I can agree to it now.

• • • • •

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"Q. And will you agree that the material picked up and sprayed through the gas contains ammonium sulphate crystals? A. I think that would be the case, because while the bath is very deep there are undoubtedly crystals at the top of the bath. I am not too sure about that, though, Mr. Brown, because I don't know exactly what this condition would be at the top of the bath, but I think there are crystals in that spray liquor.

"Q. To help you perhaps make up your mind, will you look at page three of the patent, first column, second line, where the sentence is, 'The sprayed liquor containing ammonium sulphate'---

* * * * *

"Q. I was quoting that sentence, 'The sprayed liquor containing ammonium sulphate, both dissolved and crystalline', that refers and is a statement by Tiddy that his spray did contain ammonium sulphate crystals? A. Yes.

"Q. You agree to that? A. Yes, I agree.

"Q. Will you also agree that in the apparatus, which for the purpose of my question is illustrated in the upper part of Figure 2 at the right of the drawing, the space between the roller and the casing in which the gas is subjected to spray is unobstructed? A. Yes.

"Q. In other words, sulphuric acid solution containing ammonium sulphate crystals is sprayed through coke oven gas in an unobstructed space, as I understand it? A. Yes, I would agree to that."

After this cross-examination, the plaintiff then introduced in evidence the original file wrapper in connection with one of Otto's earlier patent applications, namely, No. 659,444, in an effort to show that this Tiddy patent was mentioned as a reference and was considered by the

Patent Office. However, there is no evidence before the Court that these earlier applications which culminated in Otto's spray patent in suit were vigorously prosecuted in the Patent Office, and there is nothing in the file wrapper of the patent in suit to indicate that the Examiner considered, in any particular, this Tiddy patent.

The defendants rely also upon the use of sprays by one Underwood, who was then in charge of saturator operations at the Bethlehem, Pennsylvania, plant of the Bethlehem Steel Corporation. Crackerpipe saturators were then in use at this plant and, sometime in the year 1942 and long prior to Otto's date of invention, Underwood modified these saturators by adding spray pipes in the unobstructed space through which the coke oven gas passed above the mother liquor pool. These pipes contained holes over which were welded plates, and as the liquor was pumped through the holes and against the plates, a splash spray was created. Underwood did not rely solely upon the spray method for removing the ammonia from the gas but to assist in the complete removal after partial removal by the crackerpipe method. While it is true that these splash spray installations by Underwood were used commercially for a period of some eighteen to twenty months, they were obviously not too successful since they were removed and replaced by an Otto flare-mouth dip type crackerpipe. These Underwood installations had difficulty with "salting up" and the spraying apparatus made cleaning operations necessary at frequent intervals. It does appear, however, that reports of Underwood's spray installations and operations were widely circulated throughout the industry, although Otto denies that he had any information relative thereto even though he installed the flare-mouth crackerpipe in these Bethlehem saturators in a little more than a year after the Underwood installations were removed.

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Defendants introduced also charts showing commercial use of sprays in different locations and installations by defendant Koppers. Without going into complete detail, one such installation was said to use sprays to remove part of the ammonia at one place in the process while retaining the conventional crackerpipe to remove ammonia at another place. Also, defendant Koppers had experimented with the use of sprays alone, the spray being whipped up into a large chamber to remove the ammonia from the coke oven gas. The charts of these Koppers installations, Exhibits Nos. 27, 28, 29 and 30, were intended to support defendants' contention that the adoption of the spraying process and the abandonment of all forms of crackerpipe were a part of the evolution in the art, during which period of evolution the idea of reducing the differential pressure was being progressively carried out. Defendants' Exhibit No. 30, which shows Koppers' experimentation with the use of sprays alone, was intended by defendants to show, in addition to such use, that the experiment on this saturator was made within several months after Dr. Otto's alleged date of invention and long before the issuance of Otto's patent. The removal of ammonia from coke oven gas was accomplished, long years before Otto came to this country, by the use of the conventional crackerpipe saturator. The removal of the ammonia from the gas by that method was just as complete as by the Otto spray method and no new chemical reaction is involved in the Otto process.

Otto claimed, however, before the Patent Office other advantages and beneficial results. He claimed that his invention is characterized by a desirably low gas pressure drop, resulting in an appreciable reduction in the pumping energy required to move the gas through the saturator; reduction of over-all size of saturator and floor space requirements; low construction, operation and maintenance

costs; relative simplicity, unusually high utility and broad novelty.

Certain advantages of the spray type saturator over other types of saturators were satisfactorily shown by the evidence. A much greater quantity of gas could be passed through and treated in a spray-type saturator in a given volumetric space in the saturator; troublesome salt deposits which tended to clog apparatus were materially reduced or practically eliminated; construction, operation and maintenance costs were lowered; the over-all size of the saturator could be reduced and less floor space for saturators was required.

The recent world war in which the United States was involved caused the steel producing companies to utilize their capacity to the limit and to plan thereafter for an even greater output of steel. That required increased coke production and this, in turn, required handling of greater volumes of coke oven gas in saturators occupying available space. The chemical art had long known that an efficient way of bringing a large volume of gas into contact with the surface of a liquid is to pass the liquid in finely divided form through the gas in an unobstructed space. The record shows that, by that method, more gas can be exposed to liquid surface in a given space in a given time than by other methods. Back in 1911, Doherty, in his patent No. 997,908, pointed out the rapidity of reaction and the space-saving features incident to the pray process. During this period of expansion of the steel companies, efforts were being made by Otto, Koppers and others to reduce the differential pressure head of eighteen to thirty inches. By the time of Otto's claimed invention of the spray-type saturator, the crackerpipe types had evolved until the differential in pressure had been reduced by several inches. It was a natural evolution of the art to change the saturators to handle more coke oven

gas and it was most desirable to take care of it within the same space occupied by existing saturators because many of the steel companies have little available extra space. So the art turned to the old tool of the art, the spray method of bringing the sulphuric acid solution into contact with the coke oven gas. Otto himself admitted that the spray method of treatment of gases was old, well known to the art and a tool of the art.

In 1946, when Otto says he conceived his spray-type saturator, he had an order to install a flare-mouth crackerpipe saturator at the Lackawanna plant of the Bethlehem Steel Company. Otto sought and gained permission from the Bethlehem Steel people to install his spray saturator on the condition that the saturator be so designed that it could be converted back to a flare-mouth type in case it did not work satisfactorily. Otto testified that he was not free from doubt as to whether spray nozzles would clog up, but it appears from the evidence that the spray-type installation at the Lackawanna plant proved to be satisfactory. Within about two years following this installation, Otto sold to Bethlehem Steel and constructed a spray-type saturator at the Johnstown plant and there the spray-type replaced four of the old crackerpipe saturators. Other installations followed at Detroit, Indianapolis, Bethlehem and, lastly, three spray saturators for the U. S. Steel Corporation at Clairton. Otto has built nothing but spray-type saturators since his first installation for Bethlehem Steel.

It was stipulated by the defendants that the first spray-type saturator installed by Koppers was at the Neville Island plant of the Pittsburgh Coal & Chemical Company. This saturator went on the line in August, 1949. It was originally designed for Spraco nozzles but, during the design stage, these were changed to splash plate sprays "in order to simplify the structure and reduce both op-

erating and investment cost." The splash plate sprays did not accomplish the desired ammonia removal from the gas, and baffle plates were then added on each side of the splash plate sprays and ammonia removal was considerably increased. Pittsburgh then experimented with Spraco nozzles, which were then substituted for the splash plate sprays and the ammonia removal was thereby further increased.

Koppers had originally planned to include splash plate sprays in the spray saturator installed by it at the Woodward Iron Co., Woodward, Alabama. After construction had begun on the saturator and after the splash-type sprays had been shipped to the building site, it was then decided to install Spraco nozzles in light of the experience gained at the Neville Island plant.

In 1948 or 1949, defendant Koppers had installed for defendant Wheeling, at its Follansbee, West Virginia, plant, flare-mouth type saturators to replace crackerpipe-type saturators which had been in use since 1916. Subsequently, Koppers installed at the same plant for defendant Wheeling two saturators of the spray-type, one completed and placed in operation about April, 1952 and the other about December of 1952. During the period between these installations, Wheeling's steel capacity was being increased and new batteries of coke ovens had been built, thus requiring greater saturator capacity for removing ammonia from the coke oven gas. These two saturators are not used simultaneously as either one is capable of handling the entire gas output. However, the two flare-mouth-type saturators, which were replaced by the spray-type saturators, were used alternately and either was able to take care of the entire gas output. The change-over at Wheeling to the spray-type saturators did not change the number of men necessary as saturator operators, nor did it reduce the ground space or floor space since the

two spray-type saturators occupy substantially the same space as the ones which were replaced. The pressure drop across the flare-mouth-type saturators was between ten and fifteen inches of water, and the pressure drop across the spray-type saturators at Wheeling is about six inches. More gas is being passed through the spray type saturaters than through the ones which were replaced. The sulphate production, in relationship to the amount of gas furnished the saturator, was approximately the same in both types. The Wheeling saturators are equipped with Spraco nozzles through which the mother liquor, after makeup sulphuric acid is added and containing entrained crystals, is sprayed through the coke oven gas in an unobstructed space.

The number, placement and arrangement of sprays and the direction of the sprayed liquor into the gas flow differ in many respects from Otto. Also, in Otto's apparatus he shows the main gas reaction chamber, or absorber proper, the crystallizing tank and the acid catcher as three separate pieces of equipment. These three components are necessary to make up what might be termed a complete ammonia absorber unit. In the Wheeling installations, these three components are combined in one integral unit.

From the evidence, the Court concludes that, since the Neville Island installation, Koppers has built and sold only spray-type saturators to remove ammonia from coke oven gas. Other companies, engaged in saturator construction, have likewise gone over to the spray-type saturator. Commercial success of this type is undoubted.

Otto has granted no licenses under his patent and has brought no other suits for infringement against these defendants or any other companies installing or using the spray-type saturator. Koppers is one of Otto's most active competitors in this field.

Otto claims 1, 2 and 4 and their allowance by the Patent Office have been considered at some length, but further

discussion of these and other claims in issue appears necessary. Claim 1 describes the method of continuously producing sulphate of ammonia and the different steps involved in the process. The passing of the gas containing free ammonia into a substantially unobstructed scrubbing space is anticipated by Doherty, Becker, Bosch, Hütte, Jeremiassen and Tiddy. Contacting such gas with saturated ammonium sulphate solution containing free sulphuric acid is found in Jeremiassen and in Underwood's experiments at Bethlehem. Otto withdraws ammonium sulphate liquor to make up such solution from a body of ammonium sulphate liquor in a crystallizing space *below* said scrubbing space, but Otto's preferred form, Fig. 3, would indicate that it need not be *below*. Otto describes the contacting of the gas with the ammonium sulphate solution by spraying such solution into the gas by a whirling motion, and there is no whirling motion of the spray. The spraying by Otto is under such pressure as to disperse such solution in fine spray over substantially the whole cross-sectional area of the space. This was known to the prior art, as shown by Doherty, Becker, Bosch, Hütte and Tiddy.

Claim 2 describes a method as described in Claim 1, in which the liquor introduced into the scrubbing space entrains ammonium sulphate crystals. The evidence disclosed that the mother liquor in most commercial saturators prior to Otto contained crystals and whenever this liquor was sprayed, the spray contained crystals.

Claim 4 recites a method as specified in claim 1, in which the acid content of the liquor sprayed into the scrubbing space, adjacent such gas inlet, is about five or six per cent by weight, and in which the acid content of the liquor sprayed into said scrubbing space more remote from said inlet exceeds the first mentioned acid content. These features are mere matters of good engineering.

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Claim 5 deals with coarse sprays and fine sprays and their relative positions, but the matter of coarse and fine sprays is a matter of engineering choice. In the Wheeling spray saturator, the finer spray is nearer the outlet, which is just the reverse of the arrangement described in claim 5.

A comparison of claim 7 with claim 1 discloses much repetition, the elimination, however, of the contacting of the solution with the gas by spraying by whirling motion, and certain additional features. The added features of claim 7 provide for contacting the gas with saturated ammonium sulphate solution containing free sulphuric acid and entraining solid crystalline particles and reacting with the free ammonia to form supersaturated ammonium sulphate liquor withdrawing supersaturated ammonium sulphate liquor from the scrubbing space and desupersaturating to make up all of said solution used in carrying out the method from a body of ammonium sulphate liquor in a crystallizing space, etc. Another feature is the adding of sulphuric acid to the liquor at a rate so related to the rate at which the ammonia containing gas is passed into said scrubbing space as to effect the reaction of substantially all of the ammonia in the gas with the acid, and make up for the acid combining with ammonia in the scrubbing space.

As to the entrainment of solid crystalline particles in the liquor introduced into the scrubbing space, see the comment above respecting claim 2. The withdrawal of supersaturated ammonium sulphate liquor from the scrubbing space, and desupersaturating the same as described in claim 7, and the rate of adding sulphuric acid to replace the acid lost in combining with the ammonia, were known to the art as shown by Jeremiassen and practiced by Underwood in his Bethlehem modified saturator operations.

Claim 14 covers detailed arrangements of apparatus,

but Dr. Otto said in his deposition (page 79) that none of these details was essential.

Claim 15 adds to claim 14 nothing more than a statement that the gas supply pipe discharges horizontally into the scrubbing space and that a spray is located in this supply pipe. This construction is not found in the Wheeling saturator.

Claim 16 adds to Claim 14 a recitation of piping to permit the passage of hot water through the spray nozzles as a cleaning fluid. This was old in the art and non-inventive.

Claim 18 merely adds additional piping details to Claim 14 and the construction is substantially shown in Jeremiassen.

Claim 19 covers other details of apparatus, piping, etc., which Dr. Otto described in his deposition as non-essential. Also, this claim 19 reads substantially on Jeremiassen's Figure 1 if it should be provided with Spraco nozzles.

In reaching its decision, this Court has considered, in addition to the matters herein discussed at length, other matters mentioned briefly herein or presented at the trial. A few of such considerations are as follows: (1) The presumption of validity arising from the issuance of the patent; (2) commercial success of the spray-type saturator; (3) increased volume of coke oven gas handled by the spray-type saturator and other incidental advantages; (4) outline for lecture course given by Koppers' employees; (5) Koppers' advertising of advantages of spray-type; and (6) the many court decisions cited in the briefs of plaintiff and defendants.

The Court's attention was called to a great number of court decisions and, in many instances, the cases would appear to be not free from conflict. However, this Court has endeavored to select and to cite a number of decisions and apply them to the facts of this case. Many cases dis-

cuss invention, what constitutes it, and what is not invention. A patent is valid only if it required invention to produce its subject matter. There must be creative originality, resulting in something not available to others in the prior art to which the invention relates. Unless the claims defining a patented invention state substance not known in the prior art, the courts disregard mere verbal distinctions. Otherwise, any prior art not identical with the claim could be avoided.

In recent years the courts, under the leadership of the Supreme Court of the United States, have more and more applied the test of whether it required invention to produce the patented subject, in determining validity of a patent. If a patentee merely aggregates old elements and uses knowledge available to all workers in the art, but without any new creative mental originality of the patentee, and without producing any new and different result, such patents are held invalid even though not specifically anticipated by *any one* prior publication or use, and even though the patented subject matter may have material advantages over previously used similar subject matter, and even though outstanding commercial success is enjoyed by the patentee.

U. S. C. A., Title 35, Section 103, provides:

> "A patent may not be obtained though the invention is not identically disclosed or described as set forth in Section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains".

In Powers-Kennedy Contracting Corporation, et al. v. Concrete Mixing & Conveying Company, 282 U. S. 175, 186, the Court stated:

"For these reasons we find that the patent is invalid. It consists of a combination of elements all of which were old in the art. Its application to the transportation of concrete did not involve invention. Neither the combination of old elements or devices accomplishing no more than an aggregate of old results * * * nor the use of an old apparatus or appliance for a new purpose * * * is invention".

The Fourth Circuit Court of Appeals has repeatedly held patents invalid for lack of invention. In Garry W. Todd v. Sears, Roebuck and Company, 216 F. 2d 594, the Court found invalidity for lack of invention. At page 596, that opinion quotes from Great Atlantic & Pacific Tea Company v. Supermarket Equipment Corp., 340 U. S. 147, as follows:

"Courts should scrutinize combination patent claims with a care proportionate to the difficulty and improbability of finding invention in an assembly of old elements. The function of a patent is to add to the sum of useful knowledge. Patents cannot be sustained when, on the contrary, their effect is to subtract from former resources freely available to skilled artisans. A patent for a combination which only unites old elements with no change in their respective functions, such as is presented here, obviously withdraws what already is known into the field of its monopoly and diminishes the resources available to skillful men. This patentee has added nothing to the total stock of knowledge, but has merely brought together segments of prior art and claims them in congregation as a monopoly."

Earlier in the opinion in the Great Atlantic & Pacific Tea Company case, at page 152, the Supreme Court said:

"The conjunction or concert of known elements must contribute something; only when the whole in some way exceeds the sum of its parts is the accumulation of old devices patentable."

In Lincoln v. Stewart-Warner, 303 U. S. 545 (cited also in the Todd case), the Supreme Court said:

"The mere aggregation of a number of old parts or elements which, in the aggregation, perform or produce no new or different function or operation than that theretofore performed or produced by them, is not patentable invention."

Tropic-Aire, Inc. v. Cullen-Thompson Motor Co., 107 F. 2d 671, 674, 10 Cir., holds:

"A mere carrying forward of a known principle, or change in form, size, proportions or degree, or doing the same thing in the same way by substantially the same means, with better results, is not in itself invention."

This case was also cited with approval in the Todd case, supra.

In Young et al. v. John McShain, Inc., et al., 130 F. 2d 31, 32, the Court of Appeals for this circuit said:

"We agree with the Court below that none of the claims is valid but that all cover mere aggregations of devices, which were old in the art of steel-concrete construction, and which perform in the aggregation merely the functions which they performed in the prior art, without any new effect or resultant from their having been brought together. It is well settled that invention cannot be predicated on such aggregation of old elements even though their bringing together may have resulted in a useful product."

The Court of Appeals for the Fourth Circuit held a patent invalid for lack of invention over prior art that *suggested* what the patent in suit claimed. In Vapor Blast Mfg. Co. et al. v. Pangborn Corporation, 186 F. 2d 230, 235, the Court said:

> "And, particularly apposite in this connection, are the words of Circuit Judge Soper, speaking for our Court, in Remington Rand Business Service, Inc. v. Acme Card System, 71 F. 2d 628, 635: 'It is not necessary, however, for the purpose in view, that the Anchell patent be considered a complete anticipation to the patent in suit. It is sufficient that it suggests to one interested in the problem the means of solving it. When we consider the result which Soans was striving to achieve, and note the comparative simplicity of the problem, it is clear that it did not require invention to solve it in view of the suggestions in the kindred art contained in the Rudolph and Anchell patents.'"

W. D. Dodenhoff Co., Inc. v. Ganstonia Textile Machinery Company et al., 228 F. 2d 539, 540, 4 Cir. 1955, held a patent invalid for lack of invention over knowledge available in the prior art and suggestive of the patent in suit, and the Court said:

> "The main question is whether the construction of this apparatus involved invention. We think it does not in view of the prior art."

In examining the Otto application, the Patent Office did not cite certain pertinent prior art. For example, Tiddy patent No. 1,997,757 and Hütte's Handbook were not cited. These additional art references weaken the prima facie validity of the patent. Two decisions of the Court of Appeals of the Fourth Circuit are pertinent on this point.

In Maibohm v. RCA Victor Co., 89 F. 2d 317, 321, the Court said:

> "These two patents were not cited in the prosecution of the application for the patent in suit and, while it is true that a patent is prima facie valid, yet when defenses relied upon were not known to or considered by the Patent Office, the presumption of the validity is greatly weakened."

In Gillette Safety Razor Co. v. Cliff Weil Cigar Co., Inc., 107 F. 2d 105, 107, the same Court said:

> "Neither of these earlier patents were cited by the Patent Office when the O'Malley application was pending, and since they unquestionably constitute a most important part of the prior art, the presumption of validity arising from the grant of the patent is greatly weakened."

The presumption of validity of a patent fails when the prior art before the trial court negatives invention. One of the late cases in which this principle is applied is Metals Disintegrating Company, Inc. v. Reynolds Metals Company, 228 F. 2d 885, 3 Cir. 1956. At page 888, the Court said:

> "The District Court * * * gave no indication of the basis of its conclusion that this patent is valid except to say that the patent 'is entitled to presumption of validity.' However, we think this presumption cannot stand in the face of analysis of Ziehl's claim in relation to the teachings of Hall and the skill reasonably to be expected of a practitioner in applying and adapting the teachings and practices of his art."

See also the following decisions of the Court of Appeals for the Fourth Circuit:

Bulldog Electric Products Co. v. General Electric Co., 105 F. 2d 466;
Murdock v. Murdock, 176 F. 2d 434;
Vapor Blast v. Pangborn, supra;
Ingersoll-Rand Co. et al. v. Black & Decker Mfg. Co., 192 F. 2d 270.

Commercial success of a patented invention, even though outstanding and impressive, is of no effect where a patent is clearly invalid for lack of invention, prior use, anticipation or other defenses. This controlling principle has been consistently applied by the Court of Appeals for the Fourth Circuit in the Bulldog, Murdock, Vapor Blast and Ingersoll-Rand cases hereinbefore cited. In the Vapor Blast case, the Court held invalid a patent in spite of its commercial success. The patent covered a method of cleaning and polishing metal parts by suspending abrasive particles in a carrier liquid, circulating this mixture while keeping the abrasive in suspension in the liquid, and projecting the mixture in jets against the surface to be cleaned or polished. The patent also included claims to the apparatus for practising such a method. The prior art discussed by the Court showed that sand and similar abrasives in a liquid carrier had been used to clean and polish, and that varying types of pumps for such mixtures were known. Prior workers in the art knew that since sand tends to settle out of liquid suspension, the mixture had to be stirred or agitated to maintain a uniform relation of sand and liquid. Various devices for securing that result were known. Upon the basis of such prior knowledge, the patent was held invalid in spite of its utility and wide commercial use.

"Apropos of the shift to spray-type saturators due to increased production of coke oven gas during and as a result of the late war, it is interesting to note that the Court said in the Vapor Blast case, at page 233:

> "The demand for these wet blast machines appears to have been comparatively small until World War II, when, particularly as to the parts of airplane engines, the demand took a spirited upward jump. It would hardly be said, then, that the Eppler patent was in any sense revolutionary."

In the same opinion, at page 234, the Court said:

> "The heart of this case is the correctness of the district court's determination that the claims in suit of the Eppler patent are invalid for lack of invention under the prior art. We think this determination is clearly correct. In reaching this conclusion, we are not unmindful of two points stressed by Vapor's counsel: 'The presumptions in favor of validity arising from the issuance of the Eppler patent by the Patent Office, and the favorable evidentiary value of commercial success of the Eppler machines."

This same opinion quotes from Jungerson v. Ostby & Barton Co., et al., 335 U. S. 560, 567, as follows:

> "The fact that this process has enjoyed considerable commercial success, however, does not render the patent valid. It is true that in cases where the question of patentable invention is a close one, such success has weight in tipping the scales of judgment toward patentability. * * * Where, as here, however, invention is plainly lacking, commercial success cannot fill the void."

In the Ingersoll-Rand case, supra, the Court said at page 274:

> "It is well settled that commercial success does not in itself constitute patentability. Said Mr. Justice Jackson in Great Atlantic & Pacific Tea Company v. Supermarket Equipment Corp., 340 U. S. 147, 153, 71 S. Ct. 127, 95 L. Ed. 162: 'but commercial success without invention will not make patentability.'"

The similarity of the Vapor Blast case, supra, to this spray patent case would seem to be obvious. The "heart of this case" at bar is whether or not Otto exercised invention in spraying mother liquor with entrained ammonium sulphate crystals through coke oven gas in an unobstructed space. The teaching and knowledge of the prior art as shown by the record before this Court clearly negatives invention in so doing.

Cuno Engineering Corp. v. Automatic Devices Corp., 314 U. S. 84, found a patent void for lack of patentable invention, although the patented cigarette lighter was new, useful and commercially very successful. At page 90 the Court said:

> "We may concede that functions performed by Mead's combination were new and useful. But that does not necessarily make the device patentable • • • the device must not only be 'new and useful', it must also be 'an invention' or 'discovery' • • •. It has been recognized that if an improvement is to obtain the privileged position of a patent, more ingenuity must be involved than the work of a mechanic skilled in the art."

At page 91 of the same opinion, the Court states that the patent there in suit was invalid because and although

the patentee "merely incorporated the well-known thermostat into the old 'wireless' lighter to produce a more efficient, useful and convenient article." At page 92 of the same opinion, the Court held that the test of invention must be applied by the courts because "strict application of that test is necessary lest in the constant demand for new appliances the heavy hand of tribute be laid on each slight technological advance in an art."

In Interstate Rubber Products Corp. et al. v. Radiator Specialty Co., Inc., et al., 214 F. 2d 546, 548, 4 Cir., the Court said:

> "The touchstone is whether the new thing is beyond the ability of the worker of ordinary skill in the field, and neither the courts nor Congress have been able to offer a more definite test, so that in the end it must be left to the judgment of the courts in controverted cases to apply the formula to the specific facts; and the judges may not endow with the quality of invention every new and useful advance amidst the myriad activities of a resourceful people."

The same court in Pearson Sanding Mach. Co. v. Williams Furniture Co., 132 F. 2d 55, 58, stated:

> "A mere carrying forward of a known principle, or change in form, size, proportions or degree, or doing the same thing in the same way by substantially the same means, with better results, is not in itself invention."

In the same opinion, the Court quoted with approval a statement by the Court of Appeals of the Second Circuit as follows:

> "A mere difference or change in the mechanical construction in size or form of the thing used, in

order to obviate known defects existing in the previous devices, although those changes are highly advantageous, and far better and more efficacious and convenient, does not make the improved device patentable."

If this Court should hold valid the spray patent in suit, the effect would be to give Otto the exclusive right to remove ammonia from coke oven gas by spraying mother-liquor through the coke oven gas in an unobstructed space because Otto has stated that as the crux and essence of his invention. This, the Court cannot find. Consequently, the claims upon which the plaintiff relies in this suit, both method and apparatus, must be held invalid for lack of invention.

In view of this finding of invalidity, it would seem unnecessary to consider the question of infringement. However, the Court is of the opinion that if a finding of validity be based on the removal of ammonia from coke oven gas by spraying mother liquor through the coke oven gas in an unobstructed space, as called for by the Otto patent, then the spray saturators installed by Koppers for Wheeling in 1952 do infringe the Otto patent.

Formal findings of fact and conclusions of law as to patent No. 2,599,067 are as follows:

Findings of Fact

1. The two spray-type saturators alleged in this suit to infringe the Otto spray patent were designed and installed in 1952 by defendant Koppers, and have since been used by defendant Wheeling at Follansbee, West Virginia, in this judicial district;

2. This patent in suit describes and claims five different forms of saturators for removing ammonia from coke oven

gas. Of these five forms, only one installation as shown in Fig. 1 of the patent has been built by Otto. No saturators of the forms shown in Figs. 2 and 5 have been built. All the other saturators built by Otto under this patent have been as shown in Figs. 3 and 4, although Otto's saturators as successively installed have progressively varied in improved detail.

3. The essential feature of this Otto patent is spraying "mother liquor" containing crystals of ammonium sulphate, to which sulphuric acid has been added, through nozzles into an unobstructed scrubbing space through which coke oven gas is continuously passed.

4. "Mother liquor", drawn from a normal crystallizing pool, inevitably contains small crystals of ammonium sulphate. This has been true of all prior art saturators.

5. In the methods of spraying "mother liquor" through coke oven gas described or used prior to 1946, the liquor sprayed was obtained by drawing part or all of it from a mother liquor pool.

6. Prior art here of record shows that "mother liquor" containing ammonium sulphate crystals had been disclosed and used to remove ammonia from coke oven gas by spraying it in finely divided form through coke oven gas in unobstructed spaces.

7. Chemical engineers well knew prior to 1946 that effective contact of a liquid and a gas, for producing chemical reaction, absorption, or cooling, could be obtained by spraying the liquid through the gas in an unobstructed scrubbing space.

8. Chemical engineers well knew before 1946 that effective surface contact of a liquid and gas could be brought

about by passing the liquid through the gas in the form of a spray, shower or mist.

9. It was well known before 1946 that the finer the droplets of liquid passing through a gas, the greater the surface contact produced by any given amount of liquid, and consequently the more effective and rapid was the chemical reaction of the gas and the liquid constituents.

10. In the crackerpipe type of saturators used before 1946, coke oven gas was forced under pressure into a pool of mother liquor and passed through the pool in the form of gas bubbles, thus obtaining surface contact of gas and liquid.

11. The crackerpipe type of saturators in use for many years effectively removed the ammonia from coke oven gas.

12. No new or different chemical reaction results from the use of Otto's spray-type saturator and none is claimed.

13. The spray nozzles described in and claimed as essential elements in this patent were not invented, designed or modified in any way by Otto. He found them ready made, on sale to the public as "Spraco Nozzles", in conventional forms, adapted to produce either coarse or fine sprays. Such nozzles are described by U. S. Patent No. 1,101,264 to Eneas, issued in 1914.

14. Defendant Koppers had used Spraco nozzles such as those specified in the Otto patent in suit as early as 1926.

15. By Otto's own admissions, the spray nozzles, tar remover, acid catcher, crystallizing basin, means for controlling the size of ammonium sulphate crystals, means for continuously supplying additional sulphuric acid, means for

recirculation of "mother liquor" from the crystallizing basin to the scrubbing chamber, etc., were not original with him and were known and used in commercial saturators before 1946.

16. By Otto's own admissions, it was a matter of engineering to design an apparatus for carrying out his process of removing ammonia from coke oven gas by spraying mother liquor through the gas.

17. Some of the most pertinent prior art now before the Court was not cited by the Patent Office. (See written opinion.)

18. In 1942 the Bethlehem Steel Company, at Bethlehem, Pennsylvania (through Underwood, its employee), modified four of its five existing crackerpipe saturators by putting in perforated "spray-pipes" above the surface of the mother liquor pool in the saturators. Mother liquor containing entrained crystals was forced through holes in the spray pipes, and the streams of liquor so produced were distributed by splash plates to cause the liquor in finely divided form to pass through the coke oven gas in the unobstructed space above the pool, and thereby removed some of the ammonia.

19. The installation of the Bethlehem spray pipes was expensive, required several weeks of work of installation for each saturator. Two old crackerpipe saturators were first so equipped and put in operation. Later, two other saturators were similarly equipped and put into operation. From sometime in 1942 until 1944, the coke oven gas produced at that Bethlehem plant was treated in these combined crackerpipe and spray saturators to remove the ammonia. While these installations did not operate too satisfactorily, they were used commercially during that period.

Opinion on the Merits.

20. The splash type of liquid distribution used at Bethelehem for projecting liquid through gas was an old, wellknown and effective apparatus for producing sprays. Splash type sprays had been used in commercial saturator installations of Koppers prior to 1943.

21. The Patent Office was misled by arguments that Otto's contacting the gas and liquid was effected by spraying the liquid with a "whirling motion". Some of the claims in suit were allowed because of inclusion of that element. There is no such thing as a "whirling spray" in the sense that term was used in arguments to the Patent Office, and in the sense that such term is used in the claims in suit.

22. The two Wheeling saturators, installed by Koppers in 1952, use Spraco nozzles for spraying the liquid into the coke oven gas in an unobstructed space.

23. Other companies erect saturators of this spray type construction and use the method substantially as Otto describes in his patent.

24. Otto has no licensees under his patent and this is the first suit by him charging infringement by anyone.

CONCLUSIONS OF LAW

1. The spray patent in suit is owned by Carl Otto, the plaintiff-patentee.

2. The charge of infringement is limited to the saturators now owned and operated by defendant Wheeling, at Follansbee, West Virginia, in this judicial district.

3. There was no invention by Otto in designing in 1946 a saturator for, or using the method of, removing ammonia from coke oven gas by spraying "mother liquor", containing entrained ammonium sulphate crystals to which sulphuric acid has been added, through coke oven gas in an

unobstructed space to cause sulphuric acid and ammonia to combine in form ammonium sulphate crystals.

4. There was no invention by Otto involved in 1946 in removing ammonia from coke oven gas by the method of, or apparatus for, spraying sulphuric acid solution through conventional nozzles, with or without entrained ammonium sulphate crystals, into an unobstructed space through which coke oven gas passes.

5. The basis of plaintiff's invention, as asserted in this case, is dependent on novelty and originality with Otto of the methods and apparatus stated in conclusions 3 and 4 above.

6. Claims 1, 2, 4, 7, 8, 14, 15, 16, 18 and 19 of Otto patent No. 2,599,067 are invalid for lack of invention.

7. The prima facie presumption of validity of a patent, arising from the grant by the Patent Office, is rebuttable, and in this case has been destroyed by the evidence before this Court, in so far as the numbered claims in suit are concerned.

8. There can be no infringement of an invalid patent; therefore, the claims in suit as above numbered of Otto patent No. 2,599,067 have not been infringed by the defendants by the manufacture or use of the Wheeling saturators at Follansbee, West Virginia.

9. Defendants' counterclaim for a judgment of invalidity and non-infringement of the claims in suit of the spray patent is well founded in law, on the basis of the facts established in this record; and the judgment prayed is proper and will be ordered.

10. The complaint as to patent No. 2,599,067 must be dismissed, with costs awarded to defendants.

December 21, 1956.

Final Decree.

This action was tried before this Court, which on or about December 21, 1956, filed its opinion, findings of fact, and conclusions of law, holding for defendants; now therefore:

(1) IT IS ORDERED that the complaint filed June 14, 1954 and the amended complaint filed January 16, 1956, are hereby dismissed with prejudice, and that the defendants recover of the plaintiff their costs in this action; and

(2) That defendants' counterclaim and prayer for a judgment of invalidity and non-infringement by defendants of Otto United States patent in suit No. 2,423,794 are well founded in law, on the basis of the facts established in this record; therefore it is adjudged that the said patent is invalid and has not been infringed by either of the defendants in this judicial district; and

(3) That defendants' counterclaim and prayer for a judgment of invalidity and non-infringement by defendants of Otto United States patent in suit No. 2,599,067 are well founded in law, on the basis of the facts established in this record; therefore it is adjudged that the said patent is invalid and has not been infringed by either of the defendants in this judicial district.

Dated at Wheeling, West Virginia, this 17th day of January, 1957.

/s/ H. S. BOREMAN,
United States District Judge.

Plaintiff's Exhibit 1.

[Excerpts fom File Wrapper of U. S. Patent No. 2,599,067]

[Claim 5, as amended. This claim was subsequently cancelled. The claim is printed in outline form to facilitate study.]

The method of continuously producing sulphate of ammonia in which liquor containing sulphate of ammonia and sulphuric acid is moved along a flow path closed on itself and including an unobstructed scrubbing space and a crystallizing space, and comprising the steps of

withdrawing liquor entraining small crystals from a portion of the crystallizing space holding liquor in an approximately saturated condition

and in which small ammonia crystals are suspended,

spraying into the scrubbing space liquor and crystals so withdrawn from the crystallizing space,

passing gas through said scrubbing space, said gas when entering said space having a free ammonia content which combines within said space with sulphuric acid in, and supersaturates the liquor sprayed into said scrubbing space,

returning spray liquor supersaturated in the scrubbing space to the crystallizing space,

and agitating and recirculating the liquor in the crystallizing space to promote the growth of ammonia sulphate crystals therein and to desupersaturate the liquor returned to the crystallizing space from the scrubbing space,

withdrawing liquor-entrained relatively large crystals from a portion of the crystallizing space into which large crystals settle

and adding make-up sulphuric acid to the liquor at some point along said flow path.

Plaintiff's Exhibit 1.

Office Action September 29, 1948

* * * * * * * * * *

This Application has been examined.

* * * * *

Claims 1, 2, 3, 5, 9, 10, 12 and 16 are rejected as being substantially anticipated by Jeremiassen, cited above.

Claims 14 and 15 are rejected as being unpatentable over Jeremiassen. Any chemist in setting that process into operation would normally determine suitable gas rates and liquor circulation rates, and this would not amount to invention. . . .

* * * * *

Claims 18-26 are rejected as being unpatentable over Pyzel in view of Monsanto or Jeremiassen. No invention is seen in substituting the gas-and-liquid contact means of the latter patents for that of Pyzel. While the ultimate function of the Monsanto patent is different, the proximate function of contacting gas with liquids is the same and it is the proximate function which is controlling on the question of analogousness of art. In re Casey, 76 U. S. P. Q. 463, 609 O. G. 252.

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Amendment Filed March 28, 1949

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As has been explained to the Examiner in interviews had with him by the Applicant and by Applicant's attorney and as is made apparent by the specification of the instant application and by the records of related applications, the invention disclosed and claimed herein is of very substantial novelty and practical importance. One of the saturator constructions shown herein has been in successful commercial use since a date prior to the

filing of the instant application and is believed to have been the first saturator put into commercial use in this country or elsewhere in the world in which the small free ammonia content of coke-oven as is converted into ammonium sulphate, by spraying acidified ammonium sulphate liquor into an unobstructed scrubbing space through which the coke-oven gas is passed.

As is explained on pages 2 and 3 of applicant's specification, the saturators for recovering ammonia from coke-oven gas which were in use in this country prior to applicant's invention disclosed and claimed in the instant application, were of two types, each open to serious objections which are avoided with applicant's invention. Specifically, applicant's invention permits of the efficient recovery of the ammonia content of the coke-oven gas output of a large coke-oven plant, amounting, for example, to ninety million cubic feet of gas per twenty-four hour day, in a single spray saturator of substantially smaller bulk than one only of a plurality of saturators of the prior type heretofore required to recover the free ammonia in the gas output of such a coke-oven plant. Furthermore each of the two prior types of saturator in use in this country requires a gas pressure drop in the saturator much larger than occurs in the use of applicant's spray saturator. While the gas pressure drop in the type of saturator referred to in the paragraph beginning with line 13 of page 3 of applicant's specification is appreciably smaller than the gas pressure drop in saturators of the "cracker pipe" type, the use of the saturator referred to on page 3 involves the clogging of the spaces between the grids, requiring cleaning operations at relatively frequent intervals. Applicant's spray saturator includes no scrubbing space grids and involves no such clogging action as is inherent in the use of such grids.

Plaintiff's Exhibit 1.

The instant application is a continuation in part, of each of the five applications enumerated on page 33 of the instant application. All of those applications have been examined in Division 59. In connection with those five applications and the instant application the Examiner has cited as prior art some twelve U. S. patents, one British patent and two printed publications. The only one of those references which the Examiner has relied upon in the instant application as against applicant's broader method claims, is the Jeremiassen patent 2375922.

It is respectfully submitted that the Jeremiassen patent is not a good reference against applicant's method claims for two basic reasons, namely (1) the Jeremiassen patent contains no definite or positive disclosure of the recovery of ammonia from coke-oven gas or from any other gas having a small ammonia content; (2) the Jeremiassen patent contains no disclosure of an equivalent for the spraying action which is the primary distinguishing characteristics of applicant's invention.

There is nothing in the Jeremiassen patent which appears to be inconsistent with the view that Jeremiassen was concerned with the production of ammonium sulphate from ammonium gas or from a gas rich in ammonia such as the gas produced by the usual treatment of the "fixed ammonia" separated from coke-oven gas along with tar, as the gas is being cooled down and partially purified prior to its passage through ammonia saturator for converting the "free ammonia" in the gas into ammonium sulphate. The fact that the Jeremiassen patent does not disclose a process intended for use in the recovery of ammonia from coke-oven gas is believed to have an important bearing on the pertinency of the Jeremiassen patent as a reference against applicant's method claims. As the Examiner understands, applicant's problem was not concerned with the character of the chemical reac-

tions involved but was concerned rather with the physical conditions involved in moving a very large volume of gas, having a very small ammonia content, rapidly through a saturator of small bulk and low inherent construction cost, and operable with a small gas pressure drop. As the Examiner knows, an apparatus or method invention is not anticipated by a superficially similar prior invention, when the latter is not a full equivalent for the later invention, and was not intended to give, and is not plainly capable of giving the function or result of the later invention.

Jeremiassen does not disclose apparatus intended or operative to produce the action which is an essential and distinguishing characteristic of applicant invention. Jeremiassen regarded such a "spray" as undesirable. Thus in his patent he said that with his invention, it is not necessary

> "to resort to the use of spray or quite thin films of liquid as employed in scrubbers as this would result in formation of fine salt and coatings caused by the limit of the metastable supersaturation being locally exceeded." (Patent 2375922, p. 2, col. 1, line 74, et seq)

In fig. 1 of the Jeremiassen patent, the liquor layer above the "container 2, having a bottom provided with holes," is plainly too thin to provide the pressure head required for such subdivision of the liquid raining down through the holes, as is characteristic of the operation of ordinary spray nozzles and in particular of ordinary atomizing nozzles. As is pointed out in applicant's earlier application, Serial No. 3133, filed January 19, 1948, in commercial saturators embodying applicant's invention, the spray nozzles employed are of the widely used commercial type known as "Sprayco", anti-clogging nozzles,

embodying the invention disclosed to the Eneas U. S. Patent 101264 of June 23, 1914. Those nozzles are designed and operated to produce the sort of "spray" referred to in the last quoted statement from the Jeremiassen patent. The spray streams shown by dotted lines in fig. 2 of the Jeremiassen patent appear to be of the same non-spray type as the streams falling through discharge holes in the bottom wall of the container 2 of Jeremiassen's fig. 1.

* * * * *

Amendment Filed November 30, 1949

At an interview which applicant's attorney had with the Examiner about this case on the 14th instant, the Examiner's attention was called to reasons which were not advanced or at least were not fully explained, in the March 21, amendment paper, on which applicant relies in asserting that the Jeremiassen patent 2,375,922 is not a good reference against any of applicant's claims. In compliance with the terms and spirit of Patent Office Rule 133, the statements to the Examiner made at the interview to the Jeremiassen patent, 2,375,922 are now repeated and amplified as follows:

A fundamental difference between the invention of the Jeremiassen patent, 2,375,922, and applicant's invention is fairly indicated by the titles given the respective inventions. Applicant's invention is an improvement in the PRODUCTION OF AMMONIUM SULPHATE.

The Jeremiassen invention is an improvement in the TREATMENT OF SOLUTIONS TO OBTAIN SOLID MATTER THEREFROM. The applicant does, and apparently Jeremiassen might, treat a solution of ammonium sulphate to obtain solid ammonium sulphate crystals therefrom. Applicant's method and the Jeremiassen method are alike in that in each the solution is supersaturated as a step to the

production of solid matter, but that step by itself is not claimed to be novel by the applicant, and in fact is very much older than the Jeremiassen patents.

One broad distinction between the method which the applicant has invented and is claiming, and the method disclosed by Jeremiassen, is that *the applicant continuously makes his ammonium sulphate solution in the same chamber in which he simultaneously supersaturates the solution. Jeremiassen does not.*

While the applicant recirculates his solution through a closed circuit including the solution producing and supersaturating chamber and the crystallizing tank or chamber, the only fresh materials which the applicant needs to supply to the circulating system is sulphuric acid and coke oven gas from which ammonia is stripped as it flows through the scrubbing space. Without a substantially continuous fresh supply of sulphuric acid, applicant's process could not be carried out.

In the Jeremiassen process disclosed, no sulphuric acid needs to be supplied, and no means for supplying it are disclosed or mentioned; and Jeremiassen does not mention the composition of the "hot gas" passed into the space 4 through the pipe 5, to evaporate water out of the solution, and *that gas may well be hot air.* Jeremiassen's only reference to the addition of make up material, is his statement that

> "In continuous operation, fresh solution is constantly supplied to replace evaporated liquor and deposit solids taken up on the crystals in the suspension 11."

The apparatus disclosed in the Jeremiassen patent is apparently adapted for use just as is the apparatus comprising the associated crystallizer tank 55, evaporator 61, and circulating pump 59, shown in Fig. 4 of the Tiddy patent 2,368,901. Said Tiddy patent discloses and claims

the method of producing ammonium sulphate from coke oven gas described in the Tiddy publication referred to on page 3 of applicant's specification. It is of possible interest to note that the Tiddy publication was brought to the Examiner's attention during the prosecution of the application for the Tiddy patent which was examined and allowed in Div. 59. A copy of the Tiddy publication is included in the file wrapper of the Tiddy patent.

A characteristic feature of the Tiddy process, is that the coke oven gas is scrubbed with an acidified ammonium sulphate solution in a "packed scrubber tower" 39, which is separate and distinct from the crystallizer tank 55 and from the evaporator 61. The symbols 39, 55 and 61 are those used in the Tiddy patent. In its advertising literature, the owner of the Tiddy patent puts great emphasis on the fact that the Tidd, saturator, crystallizer and evaporator units, are separate units, each subject to the independent control. Note, for example, the paper by Frans Wethley published in the "Blast Furnace and Steel Plant", for August, 1945, and reprinted for distribution by the Wilputte Coke Oven Corp., of which Mr. Wethley was a Vice-President, and which was a wholly owned subsidiary of the owner of the Tiddy patent.

As the Tiddy publication and patent made clear, ammonium sulphate is not "made", or "produced", in the portions of the Tiddy apparatus corresponding to the evaporating chamber and crystallizing tank of the Jeremiassen apparatus, but is made in the packed scrubbing tower 39 of the Tiddy patent, for which the Jeremiassen patent discloses no equivalent. The *solution make up pipe 54* of Figs. 3 or 4 of the Tiddy patent, corresponds exactly in purpose and operative result to the *solution make up pipes 12 and 19* of Figs. 1 and 2, respectively, of the cited Jeremiassen patent. If Jeremiassen had intended to make ammonium sulphate solution in the space

4, he would have arranged to pass sulphuric acid, not sulphate solution, through said pipes, or through substitutes for those pipes.

Applicant's method differs also from that disclosed in the Jeremiassen patent, 2,375,922 in each of the following respects:

1.

The applicant entrains sulphate crystals in the liquor sprayed into the scrubbing space so that the supersaturation of the spray liquor, and its desupersaturation by the transfer of sulphate from the supersaturated liquor to the crystals, may proceed *simultaneously* in the single scrubbing space, so that the degree of supersaturation acquired by the spray liquor in the spray space may be thus reduced.

Jeremiassen does not entrain crystals in his scrubbing liquor. He wants no solid sulphate in his evaporator space 4. He expressly avoids the use of spray or quite thin films, because it "would result in formation of fine salt and coatings caused by the limit of the metastable supersaturation being locally exceeded."

2.

Applicant sub-divides the scrubbing liquor into fine spray particles while, as just noted, Jeremiassen intentionally "avoids the use of spray, or quite thin films of liquor as employed in scrubbers."

While the Jeremiassen patent seems to suggest a possibility of using the apparatus disclosed for concentrating a solution of ammonium sulphate and precipitating crystals therefrom, in such manner that the reaction of ammonia with sulphuric acid will contribute to the concentration produced, the suggestion is vague and indefinite, and does not amount to an operative disclosure of the idea suggested. The patent does not disclose or suggest any

source of the ammonia needed for such reaction, or disclose or suggest any source or means for supplying the sulphuric acid needed to react with the gas, although nearly 1500 pounds of sulphuric acid must be added to a little over 500 pounds of ammonia, to produce a ton of ammonium sulphate salt. *The Jeremiassen patent suggests no difference in composition between the solution drawn out of the crystallizer* suspension space 11 by the pump 1, and the "fresh solution" supplied through the pipe 12 or 19.

The apparatus shown in the cited Jeremiassen patent is similar in character to the crystallizer and evaporator units 55 and 61 shown in each of Figs. 3 and 4 of the Tiddy patent. The solution make up supply pipe 54 of each of those Figs. corresponds precisely to each of the make up pipes 12 and 19 shown in the Jeremiassen patent.

The fact that the apparatus shown in the cited Jeremiassen patent is similar to the crystallizing and evaporating apparatus shown in Figs. 3 and 4 of the Tiddy patent, is not surprising, since the last mentioned apparatus is disclosed and claimed in Jeremiassen's earlier patents 2,042,661, and 2,164,112. Those patents issued to the Norwegian concern named Aktieselskapet Krystal and commonly known as "Krystal". Jeremiassen was associated with Krystal for years, and assigned his applications for patent 2,375,922 to Krystal. Tiddy acknowledged in his above mentioned publication that the Tiddy process crystallizer apparatus was designed by Krystal in the following words:

> "To effect the cooling by evaporation in this process a Krystal unit, to which Semet-Solvay Engineering Corporation acquired the exclusive rights for the by-products coke industry in the United States, was chosen."

It is respectfully submitted that the one Patent Office action heretofore made in this case was unrealistic, when

considered in the light of the facts now known to the by-product coke oven industry of this country. As was explained to the Examiner at the interview, workers in that industry now regard the subject matter disclosed and claimed in the instant application as the most important development in the art of converting the ammonia in coke oven gas into ammonium sulphate, which has been made at any time within their memories. For example, as the Examiner was told at the interview, there is now in regular and successful operation at a plant of the Bethlehem Steel Company in Johnstown, Pennsylvania, one of applicant's saturators of a type and form substantially the same as that collectively shown by applicant's Figs. 3 and 3A, in which the scrubbing chamber has a diameter of 6½ feet, and is located in a corner of a saturator building, which had previously housed four cracker pipe saturators of the well known oval type and each having transverse horizontal diameters of ten and fifteen feet. Those four saturators, each having a horizontal cross section about four times as great as applicant's saturator, were designed and used to collectively recover the ammonia in the same amount of coke oven gas from which the ammonia content is now being recovered more efficiently and less expensively by applicant's one little 6½ foot diameter saturator.

None of the five references cited by the Examiner discloses a saturator intended or adapted for use in converting the ammonia content of coke oven gas, or of any other gas having only a small ammonia content into ammonium sulphate.

* * * * *

Each of applicant's claims is believed to clearly distinguish in terms as well as in substance from the reference. All of applicant's claims call for the production of sulphate of ammonia by scrubbing distillation gas, or a gas which has only a small ammonia content.

* * * * *

Plaintiff's Exhibit 1.

In addition to the broad difference, just mentioned, between applicant's method and those of the references, each of applicant's present claims includes a limitation to at least one specific feature of applicant's method or apparatus not disclosed or suggested by the Jeremiassen patent or by any other cited references.

Thus applicant's broad method claim 1 is restricted to the subdivision of the liquid sprayed "into particles having an average diameter of not more than 2 millimeters." The "drops" into which Jeremiassen expected to be produced by the "disintegration of the stream of jets" would inevitably have average diameters much greater than 2 millimeters.

* * * * *

Patent Office Action of January 20, 1950

Claims 1, 5, 10, 16, 17, 18, 19, 20, 24, 25, 26, 32, 34, 37, 38, 40, 41 and 42 are rejected as not involving invention over either Jeremiassen of record or Tiddy [2,482,643] cited. It is to be noted that Jeremiassen (particularly Figure 1) shows the general combination comprehended by the contended invention including a tank containing an elongated chamber comprising a scrubbing space for two sets of sprays at different levels between the gas inlet and the gas outlet, a crystallizing portion in the lower end of the tank with provision for recycling the liquor to the sprays.

* * * * *

Letter of July 3, 1950 Transmitting Affidavits

* * * * * * * * * * * * * * * * *

SIR:

I enclose for filing in the above entitled application an affidavit of Robert Pugh and an affidavit of Harold

Plaintiff's Exhibit 1.

Cox. The said affidavits constitute a showing of the character called for by Rule 131 (Old Rule 75) to carry the applicant's date of invention back of the filing date of the Tiddy patent 2,482,643, relied on by the Examiner in the last Office Action.

The affidavits will also be relied on in an amendment which the applicant will file on or before July 20, 1950, to show that the invention disclosed and claimed in the above entitled application is a radical improvement over the prior art and is in successful commercial use.

Respectfully submitted,

JOHN E. HUBBELL
Attorney for the Applicant

Affidavit of Robert Pugh.

IN THE UNITED STATES PATENT OFFICE

Division: 59
Applicant: Carl Otto
Serial No. 14,902
Filed March 15, 1948
For PRODUCTION OF AMMONIUM SULPHATE

STATE OF NEW YORK)
COUNTY OF ERIE) ss.:

Robert Pugh, being duly sworn, deposes and says:

I am a citizen of the United States of mature age and a resident of Orchard Park, New York. I am employed by the Bethlehem Steel Company as Coke Oven Superintendent in charge of the coke oven plant forming a part of the steel works of said company at Lackawanna,

New York, and have been so employed since April 4, 1943. Before I became Coke Oven Superintendent of the Lackawanna coke oven plant I had been the Assistant Superintendent of the plant for about 12 years.

The Lackawanna coke oven plant of which I am in charge comprises 383 coke ovens, and is capable of operation, and has been operated, at the rate at which it cokes about 8,500 tons of coal, and produces about 92 million feet of coke oven gas per day. The amount of coal coked and gas produced per day varies with conditions and particularly with the steel works coke requirements, and the average coke oven gas output per day is in the neighborhood of 80 million cubic feet per day. The term "per day", as used herein means per 24 hours.

In the operation of the Lackawanaa coke oven plant during the period in which I have been connected with that plant, it has been the custom to recover the ammonia content of the coal distillation gases in a saturator or saturators in which the coke oven gas is brought into scrubbing contact with ammonium liquor having a small sulphuric acid content. Since early in August 1947, the ammonia content of the distillation gases produced in the Lackawanna coke oven plant has been converted into sulphate of ammonia in a single spray saturator, hereinafter ordinarily referred to as the "Lackawanna Spray Saturator". That saturator was constructed by the Otto Construction Corporation of New York City in general accordance with the flow sheet marked "Exhibit A Drawing" attached hereto and made part of this affidavit. The Lackawanna Spray Saturator has been in regular and successful operation continuously since August 5, 1947, except for a few brief shut down periods, none of which were for more than a day or two.

The saturator shown by the Exhibit A Drawing, comprises a vertical tank A formed with a cylindrical upper

end portion about 8 feet in diameter, a lower cylindrical portion about 11 feet in diameter, a tapered portion connecting the two cylindrical portions, and a conical or hopper bottom portion. The vertical distance between the top and bottom of the saturator tank is about 32 feet. The Lackawanna Spray Saturator is characterized by the inclusion in the saturator tank of a substantially unobstructed gas space, and by the continuous passage of coke oven gas through, and the continuous spraying into said gas space of finely divided, or atomized, acidified ammonium sulphate scrubbing liquor and entrained ammonium sulphate crystals.

The coke oven gas is passed into the lower end of the saturator gas space through a gas inlet pipe B, and passes out of said space adjacent its upper end through two side-by-side gas outlet pipes C of which only the front one is in position to be seen in the Exhibit A Drawing. Each gas outlet pipe discharges into a corresponding one of two acid catchers D. The scrubbing liquor is sprayed into the gas space of the saturator through an upper spray nozzle 1, an intermediate spray nozzle 2 and a lower spray nozzle 3. The nozzles 1, 2 and 3 receive liquor under pump pressure through pipes 4, 5 and 6 respectively. While the spray nozzle 1 is larger in diameter than the nozzles 2 and 3, the three spray nozzles are similar in construction, each being of the atomizing nozzle type and form sold under the trade name "Spraco Center-jet Nozzles" by a well known maker of such nozzles.

The liquor sprayed into the scrubbing space through the nozzles 1, 2 and 3 collects in a liquor space within the saturator tank A below the scrubbing space and having its supper level about midway between the upper and lower ends of the lower cylindrical portion of the tank shell. In operation, liquor passes continuously away from the saturator tank A through an outlet pipe 7 into an overflow tank E. That tank has an overflow outlet pipe

8, which discharges into a mother liquor tank, not shown by the Exhibit A Drawing.

A plurality of pumps "P1", "P2", "P3", "P4", "P5", and "P6", with valved pipes connected thereto, form a pumping system for withdrawing liquor and crystals from the overflow tank E through a pipe outlet 9 opening to that tank adjacent its lower end, and through outlet pipes 10 and 11 opening to the tank at intermediate levels, and from the saturator tank A through an outlet pipe 12 in communication with the lower end of that tank. The pumping system passes liquor and entrained crystals withdrawn through the outlets 9, 10, 11, and 12 to the spray nozzles 1, 2, and 3, and to the centrifuge feed tank F. The latter passes crystals to a centrifuge dryer not shown, from which dried crystals are withdrawn for storage and use. Make-up acid is added to the liquor passing to the nozzles 1 and 2 by a make-up acid supply pipe 13 extending down into the overflow tank and discharging a stream of acid directed toward the inlet end of the outlet 10.

In regular operation, liquor and entrained crystals are supplied to the various spray nozzles at such pressures that liquor and entrained crystals are sprayed through the nozzles 1, 2, and 3 into the scrubbing space at the respective rates of about 800 gallons, 170 gallons, and 170 gallons per minute. The liquor normally discharged by each of the spray nozzles 1, 2, and 3, is saturated solution of ammonium sulphate with a free sulphuric acid content. The acid content of the liquor sprayed into the saturator A is in excess of the amount required to convert all of the ammonia in the coke oven gas into ammonium sulphate, so that the liquor collecting in the lower end of the saturator tank still has a substantial acid content. The saturated ammonium sulphate liquor sprayed into, and formed in the scrubbing space of the saturator, becomes supersaturated in that space. In the liquor spaces of the saturator and overflow tanks, the liquor moving to

the liquor outlets 9, 10, 11, and 12, forms ammonium sulphate crystals and deposits ammonium sulphate in solid form on those crystals and on the crystals received from the scrubbing space, and is thereby desupersaturated.

The Exhibit A Drawing is a flow sheet illustrating the construction and general operation of the Lackawanna Spray Saturator as it exists today, and as it was when erected and put into regular operation on August 5, 1947, except in one respect. The spray nozzle 2 and the supply pipe 5 connected to that nozzle were installed in the saturator and first put into use on December 10, 1947. The inclusion of the spray nozzle 2 in the saturator was contemplated in the original design, and the tank wall opening 5A through which the pipe 5 extends into the saturator tank A was a part of the tank structure as originally constructed. When the spray nozzle 2 and pipe 5 were installed on December 10, 1947, the pipe 5 was connected to the pumping system pipe 14, so that the latter could supply liquor and crystals to the spray nozzle 2, as well as to the spray nozzle 3.

The reference symbols A, B, C, D, E and F, the reference numerals 1 to 14, inclusive, and the showing of the spray nozzle 2 and attached portion of pipe 5, have been added by pen to the Exhibit A Drawing, and the reference symbols H, I, J and K have been added by pen to the hereinafter mentioned Exhibit B drawing in referring to the apparatus shown by those Drawings.

According to my information and belief, the Lackawanna Spray Saturator was the first of its type to be erected and put into use, and constituted an outstanding development in the by-product coke oven industry because of its relatively very great ammonia recovery capacity per unit of horizontal cross sectional area, or floor space, and because of the relatively very small drop in the pressure of the coke oven gas as it passes through

the saturator, and because of its inherently low construction, operating and maintenance costs.

The Lackawanna Spray Saturator installed and put into use in August, 1947, replaced five cracker pipe saturators of a well known and widely used type, used at the Lackawanna Coke Plant immediately prior to the installation of the Lackawanna Spray Saturator. The form and dimensions of said saturators are shown by the attached print marked "Exhibit B Drawing." The horizontal cross sectional area of each of the five cracker pipe saturators replaced in the Lackawanna coke plant by the single Lackawanna Spray Saturator, was about 178.54 square feet at the saturator liquor level. In the Lackawanna Spray type saturator, the saturator horizontal cross section at the liquor level is approximately 95 square feet. The amount of gas handled per square foot of saturator cross section at the liquor level is more than 11 times as great in the Lackawanna Spray Saturator, as it is in each of the five cracker pipe saturators shown by the Exhibit B Drawing, since the normal capacity of each of those cracker pipe saturators is about 15 million cubic feet per day, as against the more than 90 million cubic feet per day capacity of the Lackawanna Spray Saturator.

In the Lackawanna Spray Saturator, the gas pressure drop between the gas inlet pipe B and the outlet from each of the two acid catchers D is approximately equal to a water head of five inches, when gas is flowing through the saurator at the rate of 90 million cubic feet of gas per day. With only 15 million cubic feet of gas flowing through each of the five cracker pipe saturators replaced by the Lackawanna Spray Type Saturator, the pressure drop between each cracker pipe saturator gas inlet and the outlet from its associated acid catcher was approximately equal to a water head of about thirty inches. This relatively great decrease in gas pressure drop obtained when the

Plaintiff's Exhibit 1.

Lackawanna Spray Saturator replaced the cracker pipe saturators, resulted in a substantial *reduction in the pumping* energy required to move the coke oven gas through the ammonia recovery apparatus, and a resultant corresponding reduction in the cost of the coke oven plant operation.

The great excess in gas pressure drop in the old cracker pipe saturators over the gas pressure drop in the Lackawanna Spray Saturator, finds its explanation in the fact that in each of said cracker pipe saturators, the gas passed into the saturator through its cracker pipe, marked H in Exhibit B Drawing, passed out of the said pipe through submerged side-wall openings I. The latter had their upper ends at a level some 19 inches below the normal or average liquor level in the saturator. The characteristic operating feature of the Exhibit B Drawing saturators is the discharge of the gas into the bath liquor in the saturator, at some distance below the upper level of the bath, so that the gas is brought into scrubbing contact with the liquor as it bubbles up through the upper portion of the bath.

In the cracker pipe saturator shown by the Exhibit B Drawing, the gas which bubbles up through the bath liquor into the gas space above said liquor, is discharged from the saturator tank through the "Gas Outlet" indicated in the "plan" view of the Exhibit B Drawing. Each of the two vertical pipes J which extend down through the roof of the saturator tank to the lower end of the hopper bottom portions of the saturator in the Exhibit B Drawing, receives a crystal ejector which is located in the lower portion of the pipe J. Each ejector is connected to a steam supply pipe and to a discharge pipe for crystals and entraining liquor, each extending down to the ejector through the corresponding pipe J. The Saturator shell or tank wall shown in the Exhibit B Drawing is formed of cast iron lined with lead, and the cracker pipe A and verti-

cal pipes J have solid lead walls, an inch or so thick. The cracker pipe H is of loop, or horse-shoe, shape, and the two legs of the horse-shoe are mechanically connected midway between the ends of the saturator by a vertical stiffening plate K formed of lead and perforated to minimize variations in bath liquor level in the opposite end portions of the saturator, and retard the tendency of the bath liquor to surge back and forth between the two ends of the saturator.

The Lackawanna Spray Saturator tank is made of Monel metal which is not attacked by the sulphuric acid in the ammonium sulphate solution, and is not lead lined. While the cost per pound of Monel metal is substantially higher than the cost of steel, cast iron and lead, the relatively small weight and bulk of the Lackawanna Spray Saturator made it economically desirable to form the saturator of Monel metal. This is particularly true since the use of the Monel metal tank involves no such maintenance cost as is required by the relatively rapid deterioration of the lead in contact with liquor in ammonium sulphate cracker pipe saturators.

With present day construction costs, the inherent cost of reproducing the Lackawanna Spray Saturator would be substantially smaller than the inherent cost of reproducing the five cracker pipe saturators shown by the Exhibit B Drawing. The five cracker pipe saturators shown by the Exhibit B Drawing were initially installed for regular operation with only four saturators in use, the fifth saturator serving as a standby. During the period in which they were in use, the gas output of the Lackawanna coke oven plant was substantially smaller than it is now, and has been since 1947. The Lackawanna coke oven plant includes no operative standby for the Lackawanna Spray Saturator.

Plaintiff's Exhibit 1.

The "Keyplan" in the upper right hand corner of the Exhibit B Drawing, shows the outlines and spacing of the five old cracker pipe saturators Nos. 1, 2, 3, 4 and 5, and the dotted "New Saturator" circles below the old "No. 1" saturator, form the outline of the Lackawanna Spray Saturator. Preparatory to the erection of the latter, the old "No. 1" cracker pipe saturator was removed. As the Exhibit B Drawing plainly indicates, the Lackawanna Spray Saturator with a horizontal cross section at the liquor level of 95 square feet, occupies only a small corner of the space originally occupied by the five cracker pipe saturators with their aggregate cross sectional area at the liquor level, of nearly 900 square feet. The great reduction in saturator floor space made possible by the use of the Lackawanna Spray Saturator, means a substantial reduction in gas piping, gas valves, and acid catchers as well as in building space required.

The replacement of the low capacity, lead lined cracker pipe saturators in the Lackawanna coke oven plant by the Lackawanna Spray Saturator, has resulted not only in a great reduction in the bulk, floor space required, and inherent construction cost of the ammonia saturator apparatus, but has also resulted in very substantial reduction in maintenance and operating costs.

ROBERT PUGH

SWORN to and subscribed before me this 7th day of June 1950.

CLARENCE E. SPRAGUE
Clarence E. Sprague
Reg. No. 2356
Notary Public in the State of New York
Residing in Erie County
at time of Appointment

Plaintiff's Exhibit 1.

Pugh's Drawings—A and B.

(See Opposite ☞)

Note

Page 347-A is on card 6.

Page 347-B is on card 7.

Plaintiff's Exhibit 1.

Affidavit of Harold Cox

STATE OF PENNSYLVANIA }
COUNTY OF CAMBRIA } ss.:

HAROLD COX, being duly sworn, deposes and says:

I am a citizen of the United States of mature age and a resident of Johnstown, Pennsylvania. I am employed by the Bethlehem Steel Company as a Coke Oven Superintendent and am in charge of the coke oven plant known as the "Franklin Coke Oven Plant" of the Bethlehem Steel Company forming a part of the steel works of said company at Johnstown, Pennsylvania, and have been so employed since May 1, 1945.

The Franklin Coke Oven Plant of which I am in charge, is normally operated to coke about 4,000 tons of coal, and to produce about 44 million feet of coke oven gas per day. The term "per day", as used herein means per 24 hours.

In the regular operation of the Franklin Coke Oven Plant during the period in which I have been connected with that plant, it has been the regular custom to recover the ammonia content of the coal distillation gases in a saturator or saturators in which the coke oven gas is brought into scrubbing contact with ammonia sulphate liquor having a small sulphuric acid content. Since 1949, the ammonia content of the distillation gases produced in the Franklin Coke Oven Plant has been converted into sulphate of ammonia in a single spray saturator, hereinafter ordinarily referred to as the Franklin Spray Saturator.

That saturator was erected by the Otto Construction Corporation of New York City, in general accordance with the flow sheet marked "Exhibit C Drawing" attached hereto and made a part hereof, and was completed and started into regular operation in June 1949. The Franklin Spray Saturator has been in regular and contin

operation since it was first started into operation, except for a few brief shut down periods, none of which were for more than a day or two. The Franklin Spray Saturator is characterized by the fact that the space enclosed by the saturator tank is substantially unobstructed gas space, and by the continuous passage of coke oven gas through, and the continuous spraying of acidified ammonium sulphate scrubbing liquor and entrained crystals into said space.

The "Saturator" shown by the Exhibit C Drawing comprises a vertical "Saturator" tank, formed with a cylindrical body portion about 6.5 feet in diameter, and about 15.15 feet long with a conical upper end portion, and a convex bottom portion. The vertical distance between the centers of the top and bottom end portions of the "Saturator" tank is about 17½ feet.

Coke oven gas is passed into the "Saturator" tank through a lower inlet from a "Gas Inlet" pipe, and passes out said tank through an upper outlet to an "Acid Catcher". From the "Acid Catcher" the gas passes into a "Gas Outlet" pipe through which the gas passes away from the "Saturator" tank.

Saturated ammonium sulphate liquor in finely subdivided or atomized form, and entrained ammonium sulphate crystals are sprayed into the "Saturator" tank through a lower spray nozzle 1, two intermediate spray nozzles 2 and 3, and an upper spray nozzle 4. Liquor falling to the bottom of the "Saturator" tank passes from the latter through a bent, liquor sealed pipe 5 into an "Overflow Tank". Liquor and entrained crystals are normally withdrawn from the lower end of the hopper bottom portion of the "Overflow Tank" through a pipe 6 by a "Salt Pump". The latter has an outlet pipe 7 passing liquor and crystals to a "Salt Settling Tank". Liquor and crystals sprayed into the Saturator through

the lower spray nozzle 1, are passed to the latter through the pipe 7 and its branch pipe 8. Liquor and entrained crystals are withdrawn from the "Overflow Tank" at a level above that of pipe 6, through an outlet pipe connection 9 by a "Spray Pump". Normally, the latter passes the liquor and crystals withdrawn through the pipe 9 into the "Saturator" tank through the pump outlet pipe 10 and the intermediate and upper nozzles 2, 3 and 4. A "Stand-by Pump" is adapted to withdraw liquor and crystals from the overflow tank through an "Overflow Tank" outlet pipe 11 opening to said tank at about the same level as does the outlet pipe 9. The outlet pipe 12 of the "Standby Pump" may be connected to either of the pipes 7 and 10. Make-up sulphuric acid is continuously supplied through an "Acid Line" having its discharge end in the "Overflow Tank" adjacent the outlets 9 and 11.

The above quoted words, "Saturator", "Gas Inlet", "Acid Catcher", "Gas Outlet", "Salt Settling Tank", "Salt Pump", "Spray Pump", "Stand-by Pump", and "Acid Line", appear on the Exhibit C Drawing and designate corresponding parts of the Franklin Spray Saturator. Reference symbols 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, M and N mentioned above have been added by pen to the Exhibit C Drawing, for use in identifying portions of the apparatus shown by said drawing to which said symbols are applied. All of the inlet and outlet pipes connected to the "Salt", "Spray" and "Stand-by" pumps include valves and form part of a pumping system which can be used to regulate the amounts of liquor supplied to the different spray nozzles, and also to permit the "Stand-by Pump" to temporarily serve the purposes normally served by the "Salt Pump" and "Spray Pump" respectively.

Plaintiff's Exhibit 1.

The spray nozzles 1, 2, 3 and 4 are each of the atomizing nozzle type and form sold under the trade name "Spraco Center Jet Nozzles" by the well known maker of such nozzles. In regular operation, the liquor and entrained crystals are supplied to the various spray nozzles at such pressures that liquor and entrained crystals are sprayed into the saturator space through each of the nozzles 1 and 4 at the rate of about 370 gallons per minute, and are sprayed through each of the intermediate nozzles 2 and 3 at the rate of about 475 gallons per minute.

In normal operation, the liquor discharged by each of the spray nozzles is a saturated solution of ammonium sulphate with a free sulphuric acid content and entrained crystals. The acid content of the liquor sprayed into the "Saturator" tank is in excess of the amount required to convert all of the ammonia in the coke oven gas into ammonium sulphate, so that the liquor passing away from the "Saturator" tank through the pipe 5 has a substantial acid content. The saturated ammonium sulphate liquor sprayed into and formed in the scrubbing space of the "Saturator", becomes supersaturated in that space. As it moves through the pipe 5 into the "Overflow Tank", and thence to the outlets 6, 9, and 11, the liquor forms ammonium sulphate crystals and deposits ammonium sulphate in solid form on those crystals, and on the crystals received from the scrubbing space, and is thereby desupersaturated.

The Franklin Spray Saturator is of a type constituting an outstanding development in the by-product coke oven industry because of its relatively very great ammonia recovery capacity, per unit of cross sectional area or floor space, and because of the relatively very small drop in the pressure of the coke oven gas as it passes through the "Saturator", and because of its inherently low construction, operating, and maintenance costs.

Plaintiff's Exhibit 1.

The Franklin Spray Saturator put into use in June 1949, replaced four cracker pipe saturators of a well known and widely used type used at the Franklin Coke Plant immediately prior to the installation of the Franklin Spray Saturator. The form and dimensions of said cracker pipe saturators are shown by the attached print marked Exhibit D Drawing. Each of said cracker pipe saturators comprises a body portion L with flat parallel sides and semi-cylindrical end portions, and having minimum and maximum horizontal dimensions of 10 feet and 16 feet respectively. The horizontal cross sectional area of the body portion L of each of the four cracker pipe saturators replaced by the single Franklin Spray Saturator is thus more than 4 times the horizontal cross sectional area of the body portion of the Franklin Spray Saturator. The amount of gas handled per square foot of cross sectional area of the body portion of the Franklin Spray Saturator is more than 16 times as great as that of each of the four cracker pipe saturators which it replaced.

In the Franklin Spray Saturator, the gas pressure drop between the "Gas Inlet" pipe point M and the point N in the "Gas Outlet" pipe leading away from the "Acid Catcher", is equal to a water head of about 6 to 7 inches, when gas is flowing through the "Saturator" at the normal rate of 44 million cubic feet of gas per day. With the gas flowing through each of the four cracker pipe saturators shown by the Exhibit D Drawing at the rate of ten million cubic feet of gas per day, the pressure drop in the gas as it moved through each cracker pipe saturator and the associated acid catcher was equal to a water head of about thirty inches.

The relatively great decrease in the gas pressure drop in the ammonia recovery gas apparatus obtained when the Franklin Spray Saturator replaced the cracker pipe saturators shown on the Exhibit D Drawing, resulted in

a substantial reduction in the pumping energy required to move the coke oven gas through the ammonia recovery apparatus. In the Franklin Coke Oven Plant, the gas is moved through the ammonia recovery apparatus by so called "Exhausters", each of which is a turbo-blower in the gas pipe line at the inlet side of said apparatus. When the four cracker pipe saturators were replaced by the Franklin Spray Saturator, the load on the turbo-blower was so decreased that the speed of the blower was reduced to about 2560 revolutions per minute, from the speed of about 3100 revolutions required when the four cracker pipe saturators were in use. When those saturators were in use, the discharge pressure of said turbo-blower varied between 60 and 65 water gage inches. When the Franklin Spray Saturator was put into use, the turbo-blower discharge pressure was reduced to 38 to 40 water gage inches.

The great excess in gas pressure drop in the cracker pipe saturators shown by the Exhibit D Drawing, over the pressure drop in the Franklin Spray Saturator finds its explanation in the fact that the gas passing through each cracker pipe saturator is injected into the bath liquor in the lower portion of the saturator through submerged orifices O in the vertical side walls of the cracker pipe P. The latter is in the form of a flattened hollow annulus open at its under side. Gas is passed into the cracker pipe P at each end through a corresponding vertical gas inlet pipe Q. The cracker pipe orifices O are vertically elongated and have their upper ends about 19 inches below the normal or average liquor level in the saturator.

The discharge of the gas into the liquor bath maintained in the saturator, at some distance below the upper level of the bath, so that the gas is brought into scrubbing contact with the liquor as it bubbles up through the upper portion of the bath is a characteristic operating feature of the saturator shown by the Exhibit D Drawing. In each

cracker pipe saturator shown by the Exhibit D Drawing, the gas moving upward out of the bath liquor passes into and through the gas space above the bath, and out of the saturator through two "Gas Outlet" openings in the top wall of the saturator. Vertical crystal ejector pipes R extend downward through the top wall of each of said cracker pipe saturators to the bottom of the latter. Each pipe R receives a steam ejector and its steam supply and discharge pipes, only a portion of the ejector apparatus in each of the pipes R being shown in the Exhibit D Drawing.

The cracker pipe saturators shown by the Exhibit D Drawing have tank walls which are formed of cast iron and are lead lined. The cracker pipe P, "Gas Inlet" pipes Q, and crystal ejector pipes R have lead walls about one inch thick. The described use of lead in the cracker pipe saturators is due to the fact that lead is substantially more resistant to the corrosive action of the sulphuric acid in the ammonium sulphate liquor than is cast iron or steel. The Franklin Spray Saturator Tank is made of Monel metal which is not attacked by the sulphuric acid in the ammonium sulphate solution is not lead lined. The reference symbols L, O, P, Q, and R, have been added by pen to the Exhibit D Drawing.

The "Key Plan" in the upper left hand corner of the Exhibit D Drawing shows the outlines and spacing of the four old cracker pipe saturators "No. 1", "No. 2", "No. 3", and "No. 4", and the outline of the "New Saturator", and illustrates the greatly reduced bulk and floor space requirement of the Franklin Spray Type Saturator, in comparison with the aggregate bulk and floor space requirements of the four cracker pipe saturators which it replaced. The required floor space affected when the Franklin Spray Saturator was put into use, was also attended by a practically important saving in the cost of the gas piping and in valves necessarily associated with

the saturators. The replacement of the low capacity, lead lined saturators formerly used in the Franklin Coke Oven Plant, by the Franklin Spray Saturator has also resulted in a very substantial reduction in maintenance and operating costs. When the four cracker pipe saturators, shown by the Exhibit D Drawing were in use, at least two men per shift were required for the regular operation of the saturators. The Franklin Spray Type Saturator is regularly operated by one man per shift.

When the cracker pipe saturators shown by the Exhibit D Drawing were in use, bath killing operations for cleaning purposes were necessary at frequent intervals. In each bath killing operation, the liquor level in the cracker pipe saturator was raised, and this made it necessary to provide a mother liquor tank. In the regular operation of the Franklin Spray Saturator, the cleaning operations do not cause liquor to accumulate in the saturator and no mother liquor tank is now needed, or included, in the ammonia recovery apparatus in the Franklin Coke Oven Plant. In the regular operation of the Franklin Spray Saturator, water is added at short intervals to the ammonium sulphate liquor at the relatively low rate required to make up for the evaporation of water from the liquor, and the water thus added to the liquor in regular operation is normally sufficient to prevent rock salt deposits in the ammonia recovery apparatus. In regular operation the water added to the ammonium sulphate solution to make up for the evaporation is mainly supplied by a 2 in. hot water supply connection to each of the "Salt", "Standby", and "Spray" pumps. To minimize risk of rock salt deposits in the "Saturator" tank and pipe, the "Saturator" tank and adjacent portion of the pipe 5 are provided with external heating means, not shown in the Exhibit C Drawing.

Plaintiff's Exhibit 1.

The replacement of the four cracker pipe saturators by the Franklin Spray Saturator has thus substantially reduced the bulk, inherent construction cost and floor space requirement of the saturator apparatus, has simplified the operation of the saturator apparatus, has substantially reduced the pumping energy required to move the gas through the saturator, and has reduced the personnel required for the operation of the saturator apparatus.

HAROLD COX

Sworn to and subscribed before me 14th day of June, 1950.

J. R. THOMPSON,
Notary Public,
My Commission Expires Jan. 7, 1951.

Cox Drawings—C and D.

(See Opposite ☛)

Note

Page 357 - C & D are on card 7.

Plaintiff's Exhibit 1.

Argument Filed July 17, 1950

In the last Office Action, most of applicant's claims were rejected as not involving invention over either the Jeremiassen patent 2,375,922, or the Tiddy patent 2,482,643.

* * * * *

The Jeremiassen patent 2,375,922, is not a good reference against applicant's claims for the following major reasons:

1. Patent No. 2,375,922 does not disclose an operative method for converting the small ammonia content of a gas such as coke oven gas into sulphate of ammonia.

2. Patent No. 2,375,922, does not teach the injection of the scrubbing liquor into the scrubbing space in the finely divided condition which is referred to as "spray" in applicant's application.

3. Patent No. 2,375,922, does not teach the desirability or practicality of spraying crystals into the scrubbing space along with the spray liquor.

The reaction of ammonia and sulphuric acid suggested in the cited Jeremiassen patent was not intended to produce ammonium sulphate, but merely to concentrate an existing solution.

Office Action of August 3, 1950.

Claims 1, 5, 10, 11, 13, 16, 17, 18, 25, 32, 34 to 42 and 47 to 53 are rejected additionally as not distinguishing patentably over Jeremiassen. The patentee discloses a substantially similar operation as claimed wherein the ammonia-containing gas is passed upwardly in a generally counter-current flow with respect to the ammonium sulphate liquor containing sulphuric acid. Attention is directed to the upper portion of Jeremiassen's tower wherein the gas flows first both horizontally and upwardly through

a first spray 3 and then upwardly through a second spray 13. The addition of make-up acid, heating means, agitating means, pumps and the like involve obvious means to perform the desired result and add nothing patentable to the basic feature shown in Jeremiassen. Certain of these obvious features are taught in Tiddy. The formation of larger crystals from the smaller crystals in the crystallizing zones of Jeremiassen and of Tiddy is the desired objective similarly as it is applicant's operation. The separation of the larger crystals from the smaller before recycling the liquor back to the absorption is inherent to Jeremiassen. It is to be noted that Jeremiassen also discloses the desirability to carry the concentration of the liquor to a state of supersaturation, such as is necessary to bring about precipiation of the ammonium sulphate crystals. The action of the gas containing ammonia causes the saturated solution to be supersaturated to a point of metastable limits (see page 1, column 2 of Jeremiassen).

Amendment Filed February 1, 1951.

* * * * * * * * * *

Applicant's prior cracker pipe patents cited by the Examiner plainly fail to read on the disclosure of the instant application, whereas the claims of the instant application plainly do read on the disclosure of that application. The claims of the application and those of the cited prior patent of the applicant are therefore not co-extensive, and the charge of double patenting would not be tenable even if the apparatus and methods disclosed in the application in the patents were identical, which is plainly not the case.

The only scrubbing space in applicant's prior patents is the bath space, and that space is certainly obstructed by the liquid therein. The space above the bath level in each of applicant's prior patents into which washing liquid is sprayed, is not a scrubbing space and the gas passing

through the space has had substantially all of its ammonia content eliminated by its previous passage through the liquor bath into which the cracker pipe dips.

• • • • • • • • • •

Amendment Filed September 25, 1951.

REMARKS

A careful reconsideration of this application as amended is requested in view of interviews which the applicant had with the Examiner on August 1 and August 20, 1951.

After the entry of applicant's amendment filed January 30, 1951, the application contained twenty-three claims. Those twenty-three claims are replaced in the present amendment by nineteen claimed numbered 61 to 79, inclusive. Some of those nineteen claims have been copied without change from the amendment filed January 30, 1951, but the requested reconsideration of the application by the Examiner will undoubtedly be facilitated by the inclusion of all the claims in a single amendment paper.

The only claims in the present amendment which the Examiner has not held allowable or expressed a willingness to allow, are claims 70, 75 and claims 76 to 79 dependent on claim 75.

At the interview which the applicant had with the Examiner on August 1, 1951, the Examiner expressed his willingness to allow the claims 61, 62, 63 and 64 of the present amendment, in addition to the claims, 65-68 and 71-73, which the Examiner had previously found allowable.

The interview which the applicant had on August 20, 1951, with the Examiner and the Assistant Examiner in immediate charge of the application, was devoted practically exclusively to a discussion of claims 69 and 75. The Examiner declined then to act favorably on either of those claims, but intimated that he might allow claim 69 after giving it further study, and a representative of the appli-

cant was subsequently informed of the Examiner's willingness to allow claim 69.

In accordance with the requirements of Rule 133(b), the reasons and explanations presented by the applicant at the above mentioned interviews as warranting favorable action on applicant's claims, are set forth in subsequent portions of these "Remarks".

* * * * *

At the August 20, 1951 interview, the applicant explained that claim 69 is patterned after the allowable method claim 61, but omits the reference in that claim to the "whirling motion" of the spray discharged by the spray nozzle. However, the claim includes express positive limitations to the saturated condition of the ammonium sulphate solution with its sulphuric acid content reacting with the free ammonia to form supersaturated ammonium sulphate, and to the relative rates at which the sulphuric acid and gas containing free ammonia are passed into the scrubbing space so as to insure the recovery of substantially all of the ammonia in the gas; and that

The Jeremiassen patent plainly teaches the necessity of continuously adding "*fresh solution*" to maintain the process claimed as novel in the Jeremiassen patent, and plainly teaches that the effective separation of ammonia from the hot gas employed as an evaporating agent, requires that the added fresh solution be "*undersaturated*". The first teaching is clearly set forth in the first sentence of the last paragraaph beginning on page 1 of the Jeremiassen patent, and the second teaching is found in the second sentence of that paragraph.

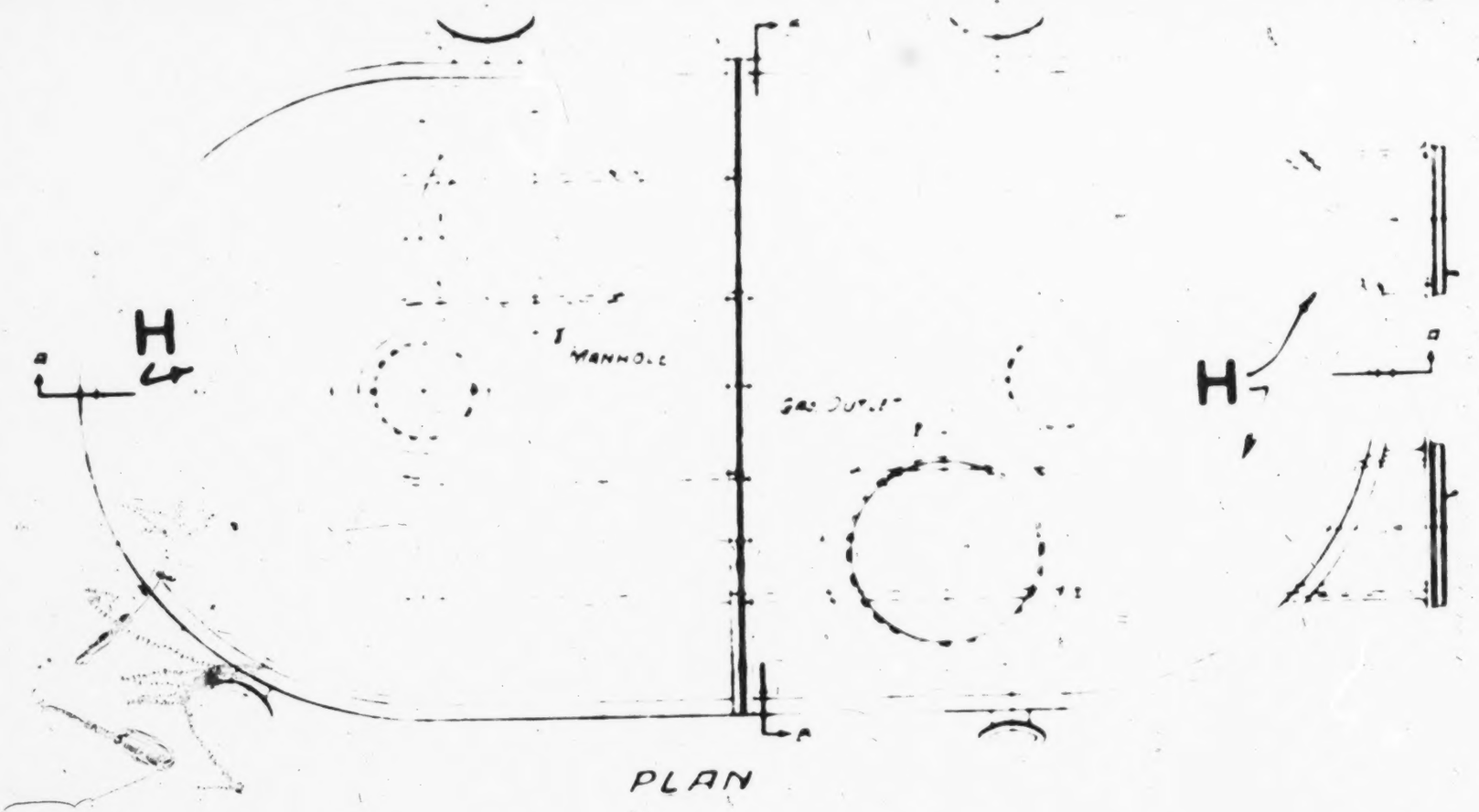
The applicant explained that the above mentioned limitations of claim 69 plainly justify the allowance of that claim, and that claim 69 should also be held allowable over Jeremiassen because the claim is directed to the essential steps of a process constituting a revolutionary advance in

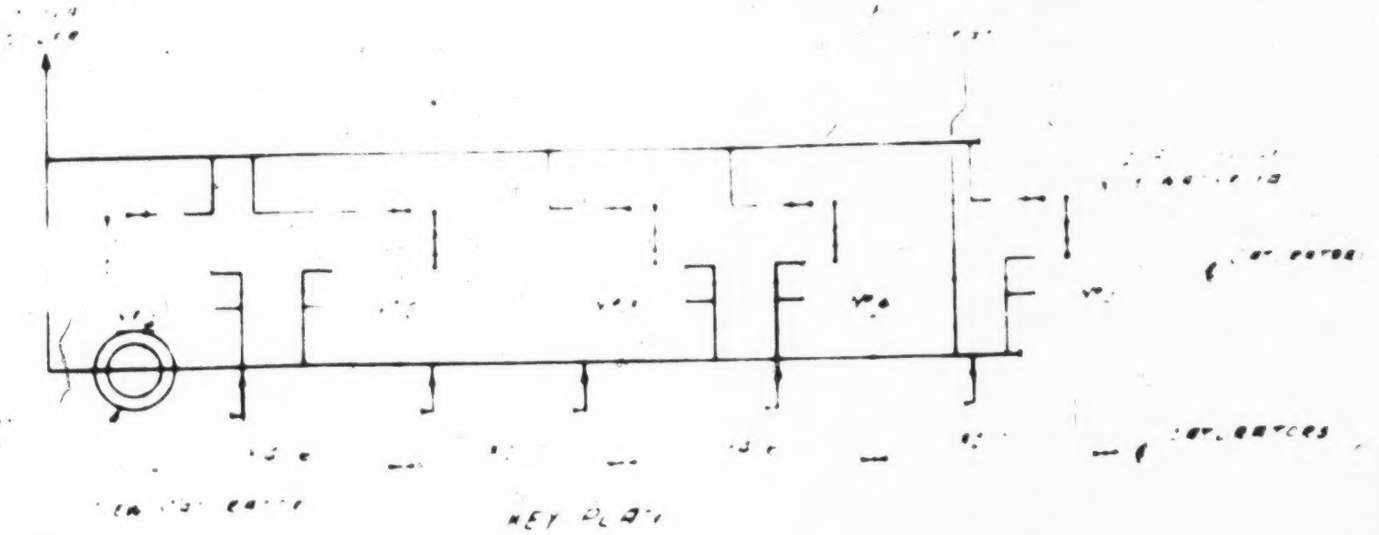
(p. 347 - A)

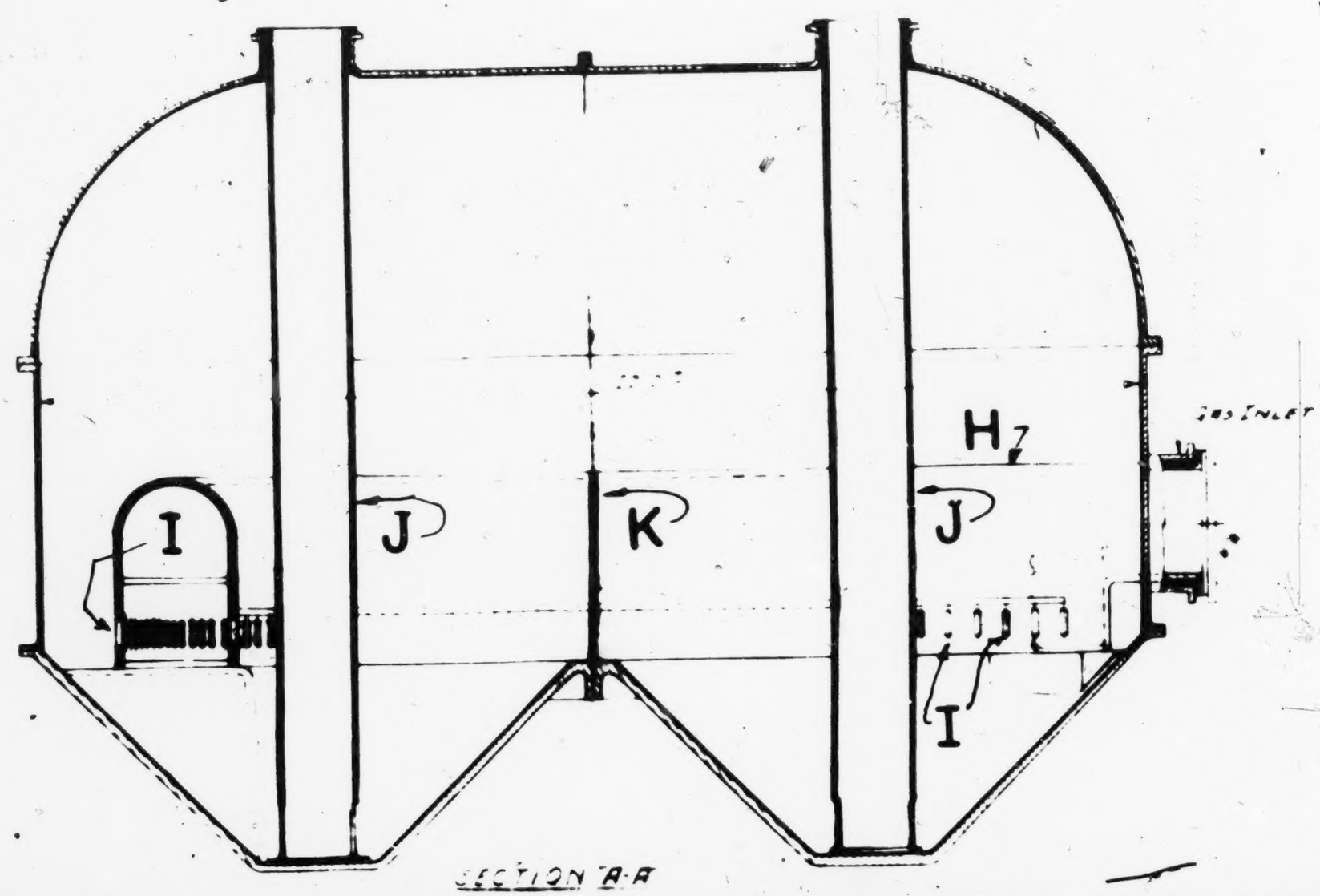
(A)

EXHIBIT A
DRAWING

(P. 347 - B)







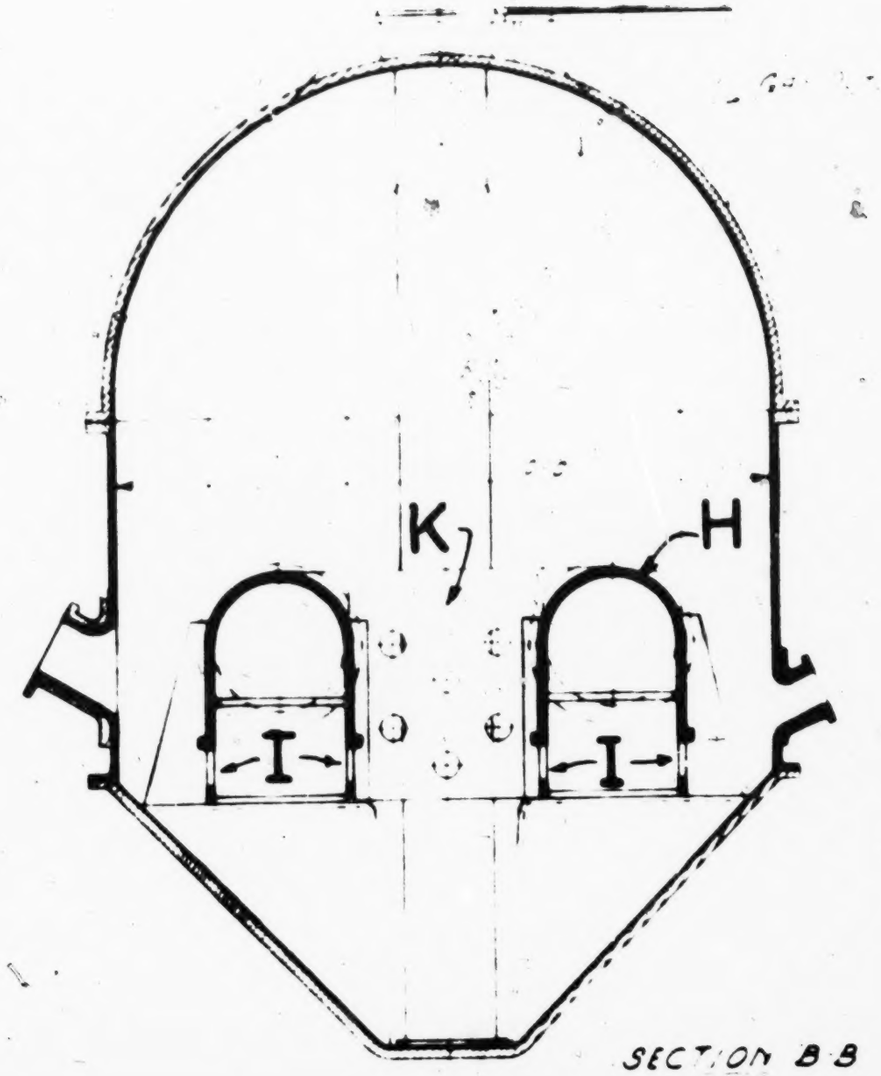


EXHIBIT B
DRAWING

(p. 275 - 1)

May 15, 1945. F. JEREMIASSEN 2,375,922

TREATMENT OF SOLUTIONS TO OBTAIN SOLID MATTER THEREFROM

Filed Sept. 15, 1939

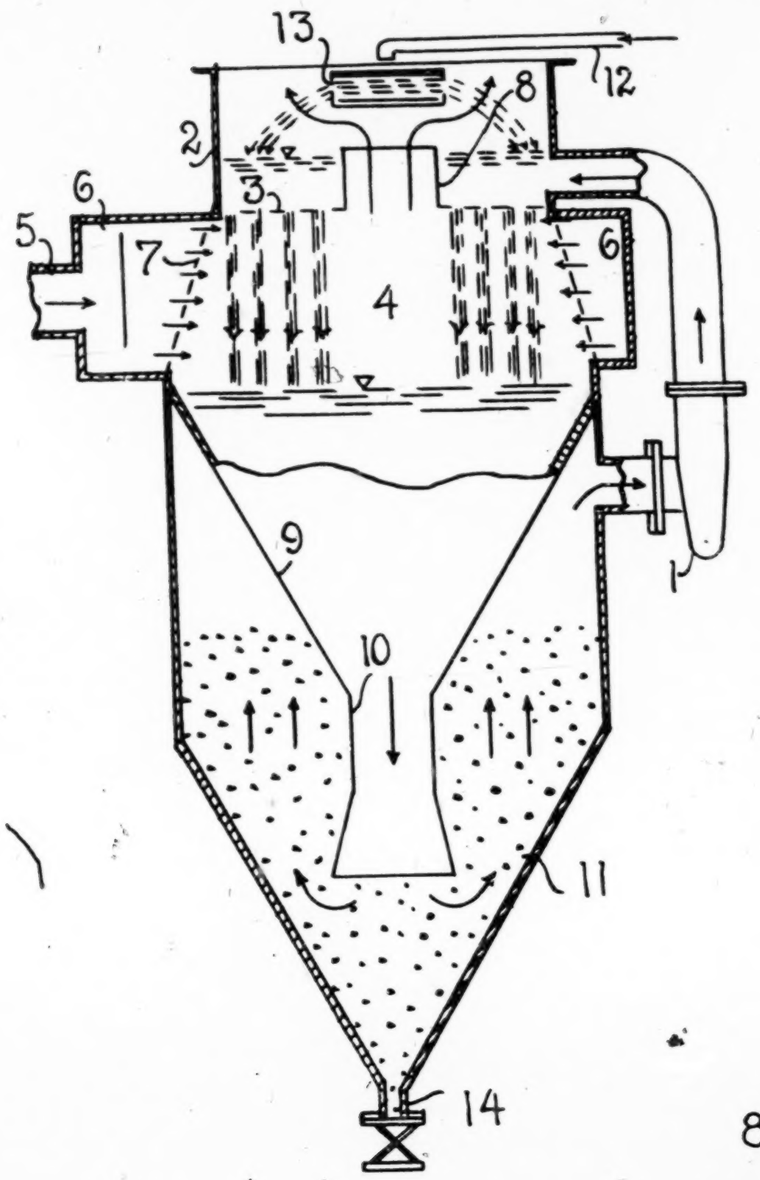


Fig. 1.

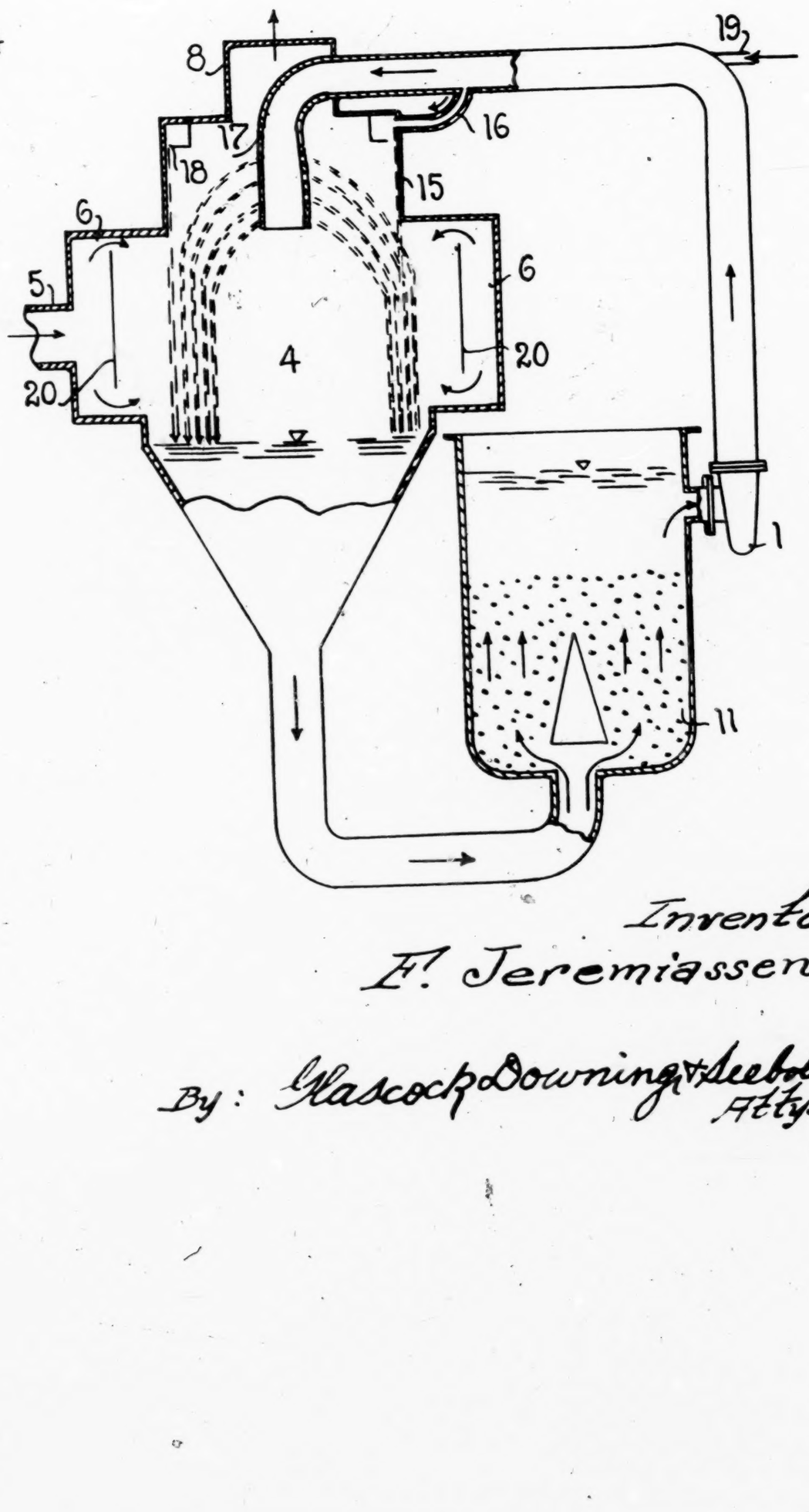


Fig. 2.

Inventor,
F. Jeremiassen

By: Glascock Downing & Seebold
Attys.

(P. 317-1)

Production of Ammonium Sulphate from Coke Oven Gas

By Frans Wethly
Vice President
Wilputte Coke Oven Corporation

WHEN the synthetic ammonia plants invaded the ammonium sulphate market with a uniform size crystal, the manufacturers of sulphate by the saturator process began to feel the effects of the competition. Saturators and accessories were redesigned, methods of operation changed, with the result that a certain amount of uniform size crystals began to be produced by the coke plants instead of the variable size and dust sulphate as theretofore. Before the competition became serious, World War II intervened; but when this war is over the coke plant sulphate manufacturers must be prepared to renew the struggle for markets.

In the following article the writer discusses the production of sulphate in saturators, in comparison with its production in the Wilputte low differential controlled crystal size process. This process has been in operation for over four years. It was devised to produce a sulphate of uniform crystalline structure instead of the unsatisfactory product of the saturator; a sulphate free-running and non-caking, comparable with the synthetic plant's best.

Crystals in ranges of sizes from small to large are produced at will by this process. This is emphasized and clearly indicated by the screen analyses cited later in this paper, varying from small to extra large crystals. The crystal sizes can be uniformly and continuously maintained to suit any particular requirement.

Saturator Operation

It is now generally known that crystals instead of fine sulphate can be produced in a saturator by providing circulation in the bottom of the saturator bath to give longer contact to the crystal so that it can grow, and by preserving a high concentration of crystal magma in the bottom of the saturator.

Circulation has been accomplished by:

(a) Propeller agitation
(b) Liquid recirculation
(c) Air agitation
(d) Gas agitation

What is to be achieved is a positive upward motion of the salt to keep the growing crystal away from the salt ejector or slurry pump suction pipe until it has grown in size to became sufficiently heavy to settle, in spite of this vertical force. In any circulation system all parts of the saturator must be kept free from accumulations of fine salt which, if undisturbed, become solid deposits of rock salt. Incomplete or localized circulation permits the building up of this rock salt in the saturator cone and in the diffuser or cracker pipe.

While difficulty due to blockage with rock salt is the chief source of trouble, erosion is an additional disturbing factor militating against operating a saturator to produce crystalline sulphate

Acid resisting tiles have been widely used to protect the lead lining of the shell of the saturator, but it is very difficult similarly to protect some of the other parts, particularly the cracker pipe. Lead is resistant to corrosion, but erosion, due to a turbulent bath carrying salt crystals, can be very pronounced, causing failure of exposed lead work. For this reason, in some installations, parts of the lead-lined equipment have been replaced by bronze alloys. The lead lining of a saturator introduces a structural weakness resulting in the necessity of frequent repairs. All saturators vibrate more or less due to the bubbling of the gas through the liquor seal. This is more pronounced when a saturator is operated below its design capacity or whenever there is poor gas distribution due to faulty design of diffusers or cracker pipe, or due to rock salt deposits. The lead lining is damaged by this vibration, being actually hammered against the shell and thereby reduced in thickness, and also being crystallized by repeated bending. In large saturators, leaks, due to lead expansion and creeping, are common.

If an agitator ring is used to create the circulation with gas or air, blockage of the holes in the ring introduces another difficulty; if circulation is increased to the extent that crystals are forced up to the cracker pipe level, increased differential, with increase in ammonia loss, cannot be avoided no matter whether the circulation is created by pump, air, gas-ring, or propeller agitator.

Further, it is important to note that the range in sizes of crystals produced in a saturator is limited. It has been established in practice that increasing the crystal size in saturators increases the gas pressure differential. This is due to higher density

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(p. 377 - 2)

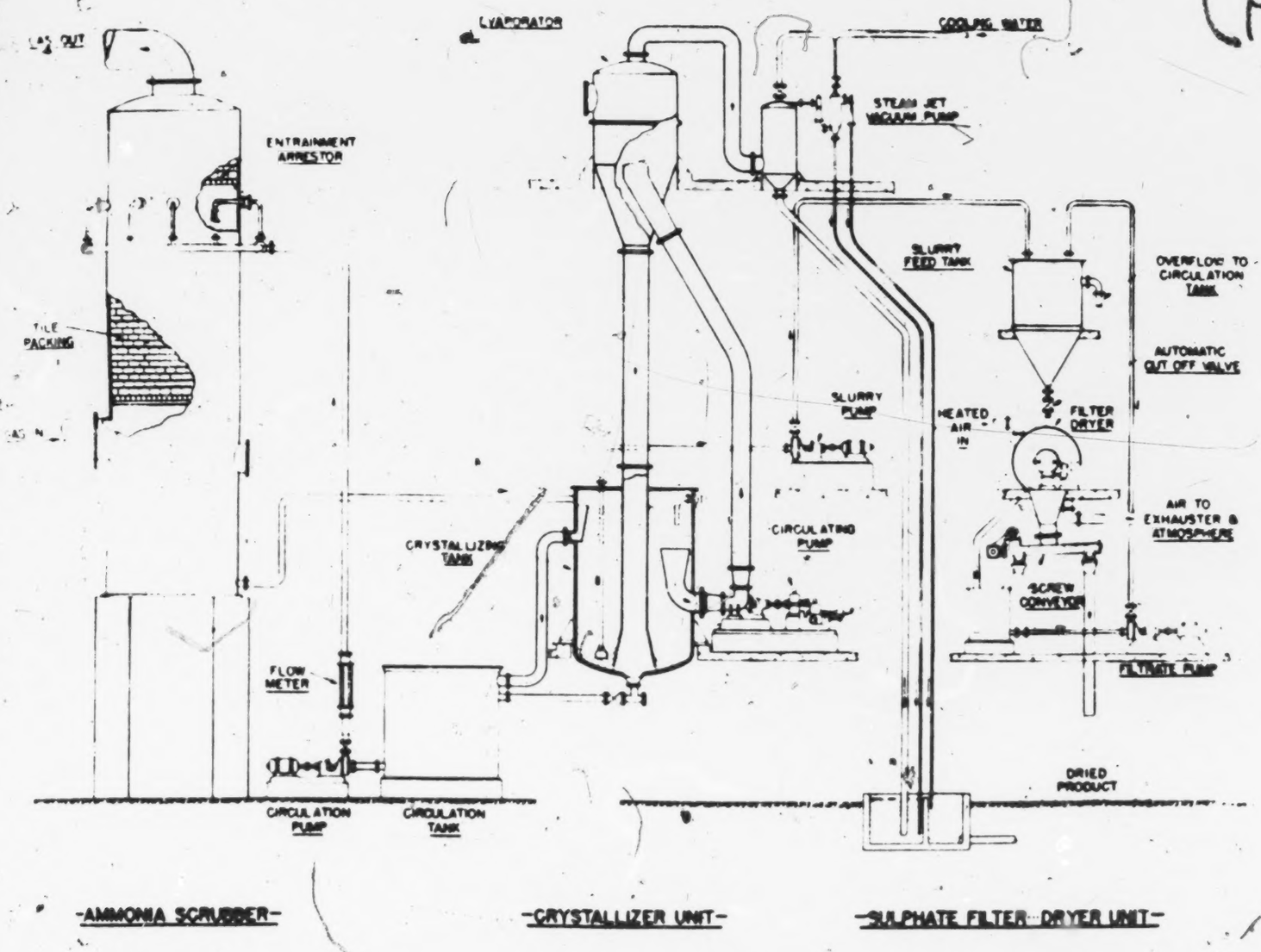


Fig. 1—Flow diagram

of the bath carrying the greater amount of crystals. Furthermore, higher crystal content of the liquor flowing over into the overflow pot, if such equipment is attached to the saturator, frequently causes blocking of this unit.

In dealing with saturator solutions, there is always danger of forming rock salt in the gas space above the liquor level. These blockages further increase the pressure differential above that caused by the gas passing through the liquor.

To remove such blockage it is necessary frequently to "kill" the bath and, after every killing, 3 to 5 tons of fine grained salt are usually produced before even small crystals are obtained.

In general, saturator difficulties have been rock salt blockage and expensive upkeep. Provided the ...om cone is so designed that it prevents accumulation of fine salt in that part and allows effective circulation, blockage occurs in the space above the bath. To reduce this blockage it is necessary to flush continuously with hot water. To evaporate the liquor resulting from adequate flushing with hot water, either the mother liquor as circulated, or the inlet gas, must be heated.

The "horseshoe" shaped cracker pipe effects poor gas distribution and liquor circulation and consequently has been found unsuitable for the continuous production of uniform crystals. A liquor seal of approximately 26 in. above the slots, equivalent to approximately 36 in. W.G. differential across the saturator, is required to effect an efficient ammonia removal.

The vertical central inlet diffuser is simpler and permits better liquor circulation. It effectively strips the ammonia from the gas with less seal depth.

With a well designed central vertical diffuser pipe, a liquor seal of approximately 12 in., causing a differential across the saturator of 18 in. W.G., after killing, suffices.

Thus it is evident that saturators, as designed up to this time, are entirely unsuitable for the continuous production of uniform crystallized sulphate.

Production by the Low-Differential Controlled Crystal Size Process

A paper by Mr. W. Tiddy, describing this process, was presented before The Eastern States Blast Furnace and Coke Oven Association, at Cleveland, Ohio, on October 24, 1941. The first plant which was put into service in the beginning of 1941 has now been in operation four years. Three additional plants are now in operation and two more are under construction.

Fig. 1 shows a flow diagram of the process. Coke oven gas is pumped by the exhauster through the tar extractor and the gas reheater, and uniting with the ammonia vapor from the ammonia still is passed through a countercurrent scrubber for the absorption of ammonia. Here it is sprayed with liquor containing approximately 5% sulphuric acid. The solution leaving the scrubber flows by gravity through a seal from the base of the scrubber to join the circulating solution in the crystallizing tank and is pumped up to the evaporator where under vacuum the sensible heat of the feed and the heat of crystallization are utilized to vaporize sufficient water to cool the solution to the required temperature. The solution is brought to the required supersaturated condition and circulated through the suspended mass of crystals in the crystallizing tank where the supersaturation is removed and the solidifying substance deposited on pre-existing crystals. The heavier crystals gravitate to the base of the crystallizing tank and are pumped to the

(p. 377 - 3)

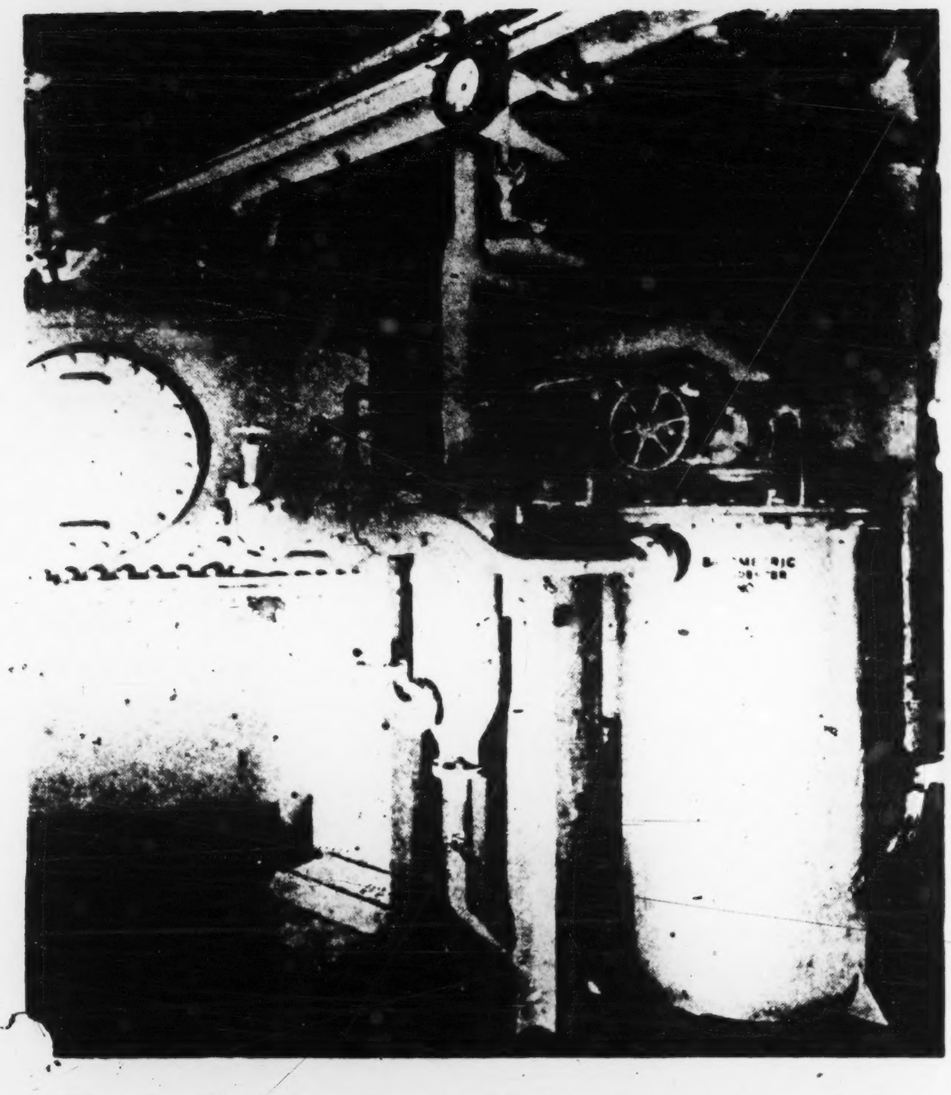


Fig. 2—Showing the top of the evaporator and the barometric condenser

slurry feed tank. From this tank, crystal sulphate is fed onto the filter-dryer where the liquor is removed and hot air under slight vacuum dries the sulphate which is then automatically discharged into storage.

Fig. 2 shows the top of the evaporator and the barometric condenser.

Fig. 3 is a view of the circulating pump with crystallizing tank and gauge board in the background.

Fig. 4 shows the filter dryer.

In the controlled crystal size process:

(a) Absorption of ammonia

(b) Cooling and concentrating by evaporation

(c) Crystallization

are accomplished in entirely separate and distinct steps as compared with the saturator process in which all three actions must take place in the one vessel.

From the very beginning when the first crystal sulphate plant was put into operation, it was evident that the process was a success. Built in the early part of the war when desired materials were difficult to obtain and substitutes had to be utilized, replacements of certain parts were required after a short operating period.

For example, the crystallizer lining was changed from lead to rubber; because the lead lining could not resist crystal erosion. The rubber lining is still in good condition after four years of service. After the operators at the first plant had sufficient time to study the continuity of operations, they removed the old saturator equipment, utilizing the free space for other purposes. They operate the plant without a stand-by unit, although they supply

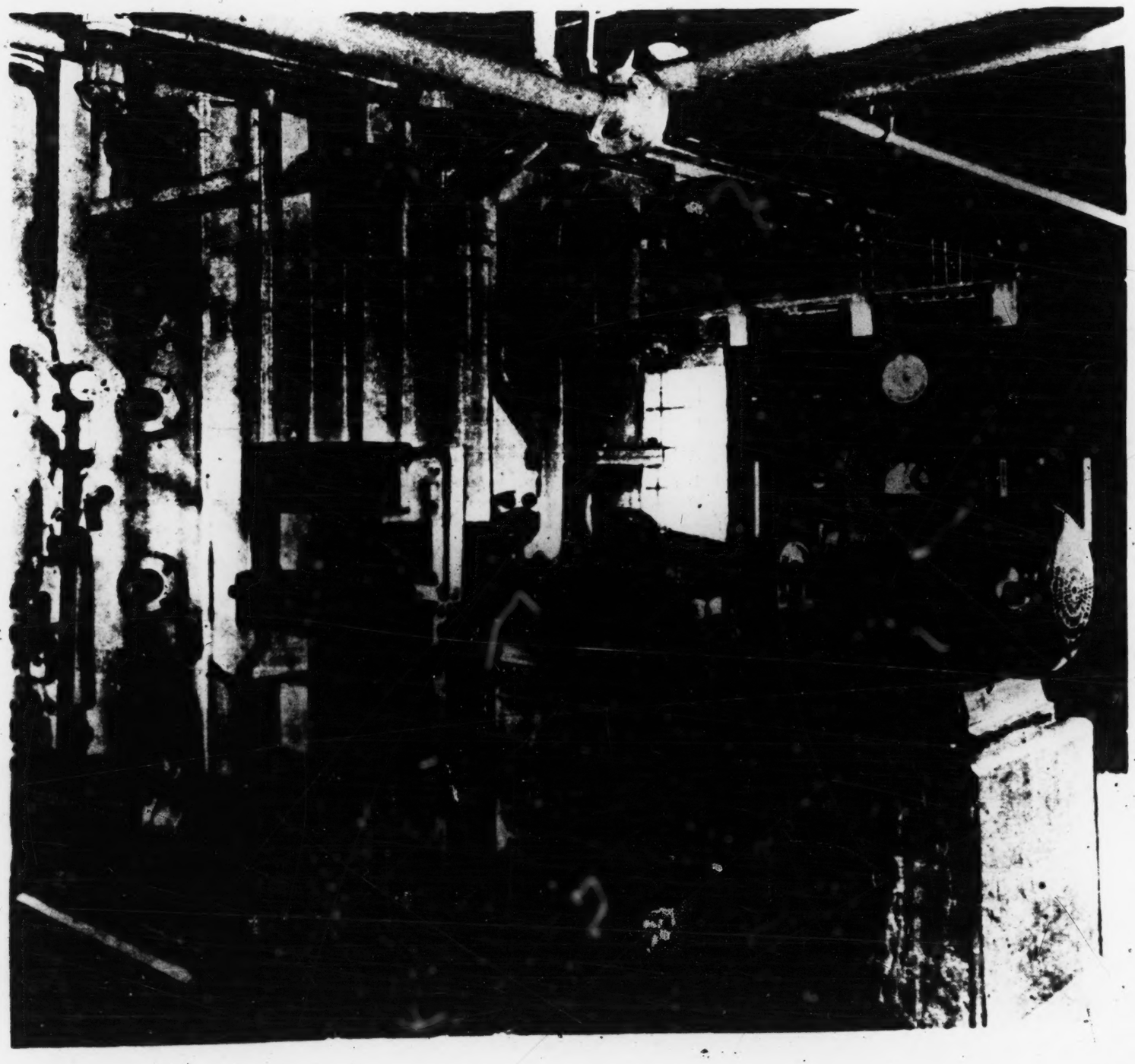


Fig. 3—View of the circulating pump with crystallizing tank and gauge board in background

(p 317-4)

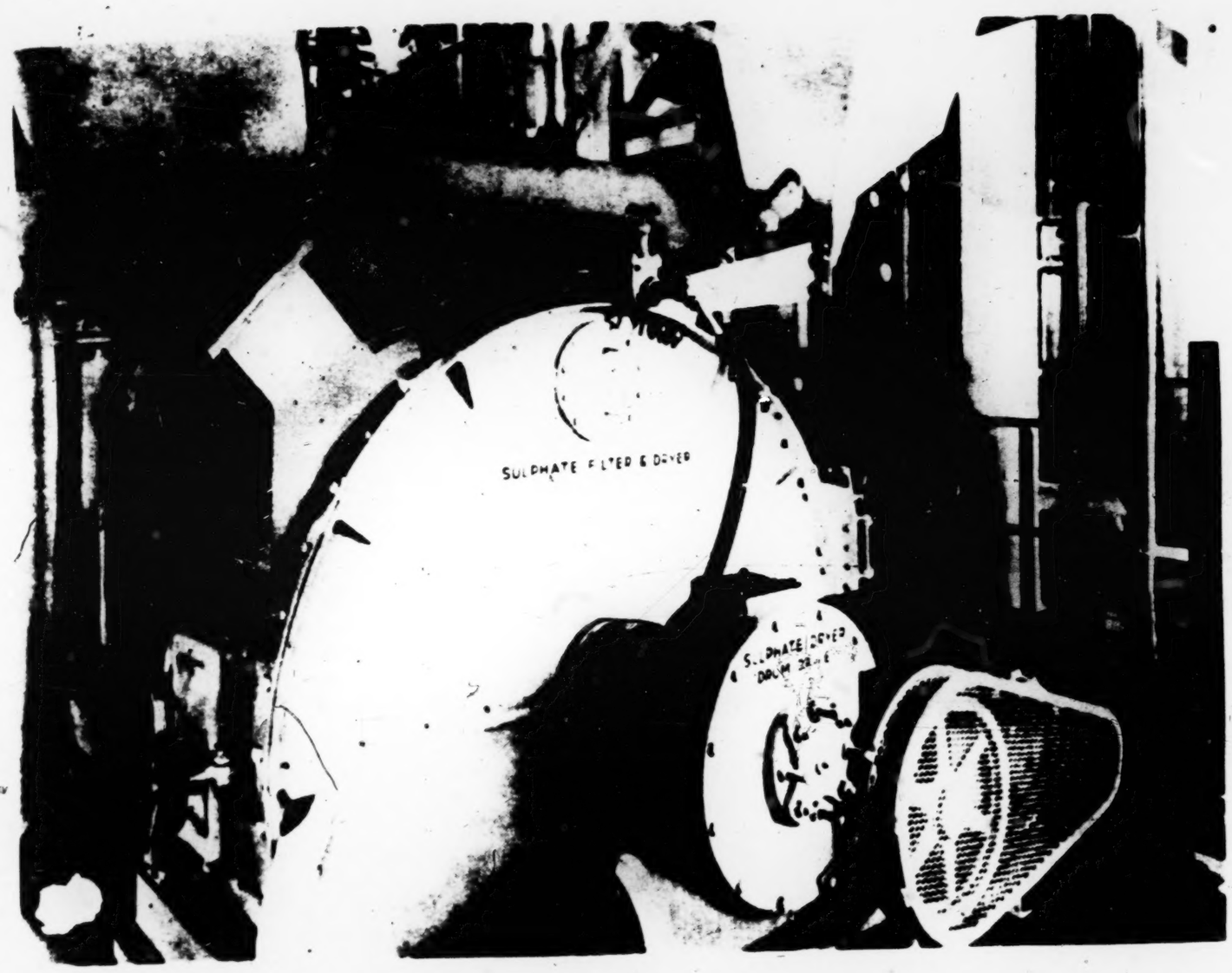


Fig. 4—Showing the filter-dryer

coke oven gas to the city. The operators at this first plant state that even with the increase in labor and material costs in 1943, as compared with 1940, the cost of sulphate production is below their 1940 costs.

Profits derived from coke oven plant ammonium sulphate production in this country, in competition with synthetic sulphate, are low, making its production hardly worthwhile, from an economical standpoint. Therefore, for maximum return, it is necessary:

(a) To reduce production costs.

(b) To produce a non-caking crystalline product which competes satisfactorily with the best grades of synthetic sulphate and which will meet the future demands of the sulphate market.

(a) Production Costs

Recovery of ammonia is effected in a simple scrubbing tower in the gas stream where constant conditions most suitable for the absorption of ammonia and for that purpose only — by a non-saturated solution, are maintained. Absorption of ammonia is 99.8%. The scrubber is lead-lined and packed with acid-resisting tiles up to a height of 12 ft. Stainless steel was considered but rejected since it was found to be insufficiently resistant to corrosion from acid solutions in the presence of coke oven gas. The top 4 feet of the scrubber, also filled with acid resisting tiles, act as an entrainment arrestor and effectively prevent carry over of the spray from the absorption section. This development in itself has eliminated the cumbersome, inefficient old type acid separator.

The lower differential through the ammonia scrubber results in a reduced power requirement of approximately 155 hp. at the exhauster turbine of a plant handling 40,000,000 cu. ft. of gas per day. This is a substantial steam saving.

The differential is uniform and is not subject to severe fluctuations, as is the case with a saturator. With two or more scrubbers in parallel in the gas stream, this steady differential allows uniform distribution of gas over several scrubbers. This is not possible with two or more saturators in parallel and, therefore, unless there is a separate exhauster for each saturator, the gas distribution is unsatisfactory, resulting in ammonia losses. The size of the scrubber is relatively small. One of 10' 6" diameter by a total overall height of 36' 0" is ample for a sulphate production of about 50 tons per day.

As shown in the flow diagram, there is a circulation tank between the ammonia scrubber and the crystallizing tank. While scrubbing of the gas is continuous, the circulation tank permits interruptions in the operation of the evaporation-crystallization unit. Thus the ammonia scrubber operation is separate and distinct from the sulphate crystal production, so that the optimum conditions can be maintained in each of these operations. This arrangement affords the desired flexibility, which is entirely missing in saturator operation. It permits the omission of complete stand-by units, thus reducing investment costs and fixed charges. The crystallizer, as well as the piping or body of the apparatus with which the crystals come in contact, is rubber lined. This has proven very satisfactory. The top section of the evaporator is of stainless steel, there being no coke oven gas present. Even though the oldest installation is in a plant with P and A tar extractors, there is no trace of attack on the rubber lining by tar or aromatic compounds.

(p. 377-5)

Rubber lining has long proven successful also in crystallizing units of synthetic ammonium sulphate plants. The Worthite pumps and rubber-lined valves give lasting service.

The steam requirement for the process is approximately one-half that for the saturator process; water and electric power requirements are slightly greater, but these are more than offset by the saving in steam effected by the exhauster delivering gas against a much lower pressure.

Upkeep and maintenance with the Wilputte process are reduced to a minimum. Complete stand-by units are not required.

Labor is reduced to the minimum. One man per shift performs all operations in connection with ammonia absorption and sulphate production, lime handling with ammonia still and prelimer, and pyridine removal equipment. The operating conditions are simple. The operator at one of the latest plants even finds time to handle the crane for the transporting of sulphate to storage.

The filter-dryer, revolving at approximately one rpm., separates the mother liquor from the crystals, including washing or neutralizing, and drying with hot air. This filtering and drying operation takes the place of the centrifugal dryers and of the large kiln or hot air drum dryer, and thus effects a material saving in operating and maintenance costs.

No sulphate pit is necessary with this equipment as the sulphate can be mechanically moved or conveyed to storage by air.

(b) Producing a Product Which Will Meet Future Demands

Either small or large crystals can be produced at will, as the crystal size is dependent upon the circulation of the solution in the crystallizing tank and the amount of crystals carried in suspension therein. Typical screen analyses of crystal size continuously produced with this process are:

	Small	Medium	Large	Extra Large
On 10 mesh	0.38%	0.05%	8.7%	12.0%
—10 + 20	12.82	22.45	59.0	76.9
—20 + 40	72.45	64.70	26.8	10.6
—40 + 60	11.32	10.80	5.2	0.5
—60 + 80	2.23	1.65	0.3	0.0
—80 + 100	0.65	0.27	0.0	0.0
—100	0.15	0.08	0.0	0.0

In producing a uniform crystal salt, a substantial amount of sulphate is in suspension in the crystallizer. In a saturator, a corresponding amount would readily result in blocking up the saturator. Even if a saturator's bottom cone were of ample proportions, the same products could not be obtained, since absorption, concentration and crystallization take place uncontrolled within the one vessel.

The sulphate made by this new process and represented by the above screen analyses is free-[illegible]ing, does not cake in storage and [illegible] produced, does not stain or injure burlap or paper bags used for shipping purposes. The free acid content of these products is from 0.05 to 0.08%. However, a neutral product is made readily if required, the only operating change being the use of a weak ammonia solution in place of the water wash on the filter-dryer.

The moisture content, of the salt from the top feed filter, is between 0.05 and 0.09%. The ammonia content is from 25.64 to 25.71%.

The market for sulphate as a top dressing which in the past has been restricted because of the large proportion of fines and dust in the saturator products, now becomes open to the crystal sulphate. This product can be distributed much more readily, can be spread evenly over greater distances, is less affected by wind and has less tendency to stick to foliage and burn it. When shipped in bulk, the losses are reduced. Manufacturers making mixed fertilizer have found that the Wilputte crystal sulphate is much easier to handle throughout their operations. Some have already expressed a preference for this product.

The sulphate from the Wilputte controlled crystal size process competes satisfactorily with the best grades from the synthetic ammonia plants and so places the coke plant producers in a favorable position to meet the demands of the market after the war. At that time we may expect that the large production of crystal sulphate from the synthetic plants will make it more difficult to market sulphate not in crystal form.

★ ★ ★

American Foundrymen's Association Elects Officers

NEW officers and directors of the American Foundrymen's Association were elected at a 1945 Administration Congress held July 17-19, at the Palmer House, Chicago. The three-day meeting, held on an invitational and delegate basis in accordance with ODT regulations, brought together both national and local leaders of the Association for framing policies and activities on behalf of the foundry industry during the coming year.

The following national officers and directors were elected:

President—F. J. Walls, International Nickel Co., Detroit.

Vice President—S. V. Wood, Minneapolis Electric Steel Castings Co., Minneapolis.

Directors—George K. Dreher, Ampco Metals, Inc., Milwaukee; E. W. Horlebein, Gibson & Kirk Co., Baltimore, Md.; H. H. Judson, Gould Pumps, Inc., Seneca Falls, N. Y.; James H. Smith, Accessories Group, General Motors Corp., Detroit; F. M. Wittlinger, Texas Electric Steel Casting Co., Houston, Texas. In addition, retiring President R. J. Teetor, Cadillac Malleable Iron Co., Cadillac Mich., was elected a director to serve one year.

the recovery of ammonia from coke oven gas, as is evidenced by the Pugh and Cox affidavits which have been filed in this case, and give results not capable of attainment with the Jeremiassen method or apparatus. The applicant also called attention to the comments on the Jeremiassen patent on pages 20 and 21 of the amendment filed January 30, 1951.

* * * * * * * * *

The arguments, explanations and reasons made and stated by the applicant in the course of the August 1, 1951 interview, were substantially entirely concerned with proposed amendments of former claim 1 and with the claims 61, 62 and 63 of this amendment paper. At that interview the applicant discussed the effect of proposed limitations to claim 1 in respect to

(1) the dispersal of the liquor "throughout the whole cross sectional area of the scrubbing space", and

(2) With respect to the supply of liquor under pressure to form "a fine liquor spray".

The applicant then pointed out that the dispersion of the liquor throughout the whole cross section of said scrubbing space was not taught by Jeremiassen, was not wanted by him, and was not obtainable with the apparatus disclosed by Jeremiassen when used in the manner disclosed by Jeremiassen, and necessary to obtain the results which Jeremiassen sought to obtain. Jeremiassen diagrammatically shows in Fig. 1, the liquor issuing from the cup 13 in the form of a section of a hollow sphere, which Jeremiassen refers to as a "'veil' of liquor" through which "the escaping residual gas from pipe 8 must pass through". The vertical streams or jets passing through and downward away from the holes in the bottom wall 3 also form an annular "veil" through which the gas supplied

by the pipe 5 must pass as indicated by the arrows extending through the cone 7.

The dotted line showing of the liquor passing away from the cup 13 and downward from the holes in the bottom wall 3 cannot be disregarded. It is a part of Jeremiassen's disclosure expressly intended by him to show what he had in mind when he specified,

> "passing the added fresh solution in the most intimate contact possible with the escaping residual gas as diagrammatically illustrated in the drawing". (Underlining ours), Jeremiassen Patent, p. 2, col. 1, lines 4-7.

The flow of the liquor in streams or jets as illustrated by the dotted lines was intentially sought by Jeremiassen not only to provide the "veils" through which the gas was compelled to pass "in the most intimate contact possible", but also to carry out Jeremiassen's idea of a voiding objectionable supersaturation which he considered inherent in

> "the use of spray or quite thin films of liquid as employed is scrubbers". (Jeremiassen patent, p. 2, col. 1, lines 74-5).

The downflow of the liquor in the streams or jets diagrammatically illustrated, was also regarded by Jeremiassen as necessary to avoid rock salt formations caused by the contact of the falling liquor with solid bodies and particularly with the scrubbing space walls. Jeremiassen seems to have intended and expected that the only contact of the liquor with those walls would be due to the "splashes" and "splashing" referred to in lines 16-17, column 2, page 2 of Jeremiassen's patent. The "splashes" and "splashing" referred to by Jeremiassen implies a liquor condition or action which does not exist when saturated ammonium sulphate is sprayed

> "under such pressure so as to disperse said solution in fine spray over substantially the whole cross sectional area of the scrubbing space",

as is specified in claim 61 which was drafted during the August 1, 1951 interview. At that interview, applicant pointed out that definiteness in respect to the pressure at which the liquor is sprayed into the scrubbing space is important only because the applicant uses pressure to form *a fine spray*, and thereby differentiate applicant's use of a fine liquor spray from Jeremiassen's non-spray streams or jets.

It is well recognized in the patent law that precision in specifying characteristics of well known elements of an invention is unnecessary, and applicant pointed out that for a generation at least, it has been common practice of numerous manufacturers of spray nozzles to tabulate the pressures to be used with different nozzles to produce coarse and fine sprays. Such tabulations are shown for example, in the Spraco catalog which the applicant showed to the Examiner and his Assistant.

Spray nozzles had been in extensive use for at least half a century before Jeremiassen's application was filed, and it was a matter of common knowledge that, other things being equal, the liquid sprayed had to be put under higher pressure to produce a fine spray than to produce a coarse spray.

* * * * * * * * * *

In the August 1, 1951 interview, the applicant called attention to the important distinctions between applicant's process and that disclosed in the Jeremiassen patent which are discussed in detail in the "Remarks" of applicant's amendment filed in the Patent Office on January 30, 1951, and directed attention particularly to pages 20, 21 and 22 of the last mentioned amendment, and called attention to

the important fact that the *Jeremiassen patent does not suggest or provide means operative to effect the production in the scrubbing of all of the ammonium sulphate solution which is supersaturated in that space.*

In the August 1, 1951 interview, the applicant commented on the suggestion that even though applicant's fine liquor spray cannot be produced with the apparatus shown in Fig. 1 of the cited Jeremiassen patent, the apparatus shown in Fig. 2 of the patent might possibly be operative with a pump delivery pressure high enough to obtain applicant's finely divided spray by Jeremiassen's discharge of liquor through the holes in his pipe 17.

In reply to that suggestion, the applicant said, in effect, that it seemed illogical to contend that Jeremiassen discloses a fine spray in Fig. 2, which he cannot obtain with his Fig. 1 apparatus, and which he plainly does not want with either apparatus. In the first and second paragraphs in column 2 of page 2 of his patent, Jeremiassen plainly indicates that the Fig. 2 apparatus, like the Fig. 1 apparatus, is intended to cause the liquid "to fall freely down through the space through which gas is flowing". *Liquid streams which "fall freely down" are not equivalent to spray particles moving under kinetic energy imparted to them by the nozzle expulsion pressure.*

* * * * * * * * * *

At the August 1, 1951 interview, it was pointed out by the applicant that his practice of dispersing the liquor passed into the scrubbing space by a whirling motion as specified in claim 61 [claim 1 of the patent], is readily effected by the use of spray nozzles of standard forms, and in particular by the spray nozzles of the well known type disclosed in the Eneas patent 1,101264.

In this connection, the Examiner's attention is invited to the following statement in lines 8 to 15 of page 24 of applicant's original specification:

Plaintiff's Exhibit 1.

> "In practice, the difference between the level of the nozzle 67 and the level of the nozzle 7b may be of the order of ten or fifteen feet, so that the pressure at which the liquor is supplied to the nozzle 67 exceeds the pressure at which the liquor is supplied to the nozzle 7b by several pounds per square inch. This difference in the supply pressures facilitates the maintenance of the desired difference in the size of the liquor droplets discharged by the nozzles at the different levels."

The pressure head due to a 10 or 15 feet column of liquor having a density of the order of 30 percent greater than the density of water, amounts to some 6 to 9 pounds per square inch, as the Examiner will recognize. Thus in the operation of the apparatus shown in Fig. 3 of his application, the applicant contemplated the pressure of the liquor sprayed through the nozzle 7b to be high enough to create a coarse spray, and that the pressure of the liquor discharge through the nozzle 67 should be about 6 to 9 pounds greater than the pressure needed to produce a coarse spray.

The foregoing references to the statements made by the applicant at the August 1, 1951 and August 20, 1951 interviews are based on the applicant's present recollection refreshed, as the Examiner will understand, by reference to draft amendments which the applicant had with him at the interviews and which the applicant had helped to prepare.

* * * * * * * * * *

Office Action October 11, 1951.

"Claim 61 defining the particularly whirling motion of the finely divided spray droplets stated to give an improved contacting between the crystal containing liquor and the gas being treated and thereby an improved yield is considered to be allowable as are the dependent claims 62-64 [claims 2-4 of the patent]."

* * * * *

Plaintiff's Exhibit 1.

Claim 69 [claim 7 of the patent] is rejected over Jeremiassen in that the limitation of spray pressure broadly stated defines no invention over Jeremiassen's gravity head pressure. The limitation regarding the recycle with entrained crystals is not considered a patentable one.

Applicant's Brief Before Board of Appeals.

* * * * *

In the commercial use of his invention, the applicant uses spray nozzles of a well known type formed to discharge spray liquid with a whirling motion. While the applicant prefers to use such nozzles, there are other well known forms of spray nozzles which do not discharge spray with a whirling motion.

It is respectfully submitted that a limitation to the discharge of "fine spray" with a "whirling motion" is plainly not essential to the patentability of claim 69 [claim 7 of the Otto patent] over Jeremiassen; and that the entrainment of crystals in the spray liquor is a patentable feature of claim 69.

Aside from its "fine spray" and crystal entrainment features, claim 69 contains limitations to two features, each of which is believed to patentably distinguish applicant's method from the disclosure of the Jeremiassen patent; namely, the feature of

> "spraying under such pressure as to disperse said solution in fine spray over substantially the whole cross sectional area of the space"; and

The feature of

> "adding sulphuric acid to the liquor at a rate so related to the rate at which the ammonia containing gas is passed into said scrubbing space as to effect the reaction of substantially all of the ammonia in the gas with the acid, and to make up for the acid

combining with ammonia in the scrubbing space", with the resultant formation in the scrubbing space of *all of the ammonium sulphate used in maintaining applicant's process for producing ammonium sulphate from coke oven gas.*

Examiner's Statement Before Board of Appeals.

* * * * *

Invention has been conceded in the process claims 61-64 [claims 1-4 of the patent] and 68 which define the whirling motion of the spray droplets as stated to give improved yield as expressed for example in claim 68 which recites the spraying at three separate levels with a different type spray at the several levels giving the improved contacting as stated.

* * * * *

Patent No. 2,599,067.

Attached to Plaintiff's Exhibit 1.

(See Opposite ☛)

(p. 361-1)

June 3, 1952 C. OTTO 2,599,067

PRODUCTION OF AMMONIUM SULFATE

Filed March 15, 1948 5 Sheets-Sheet 1

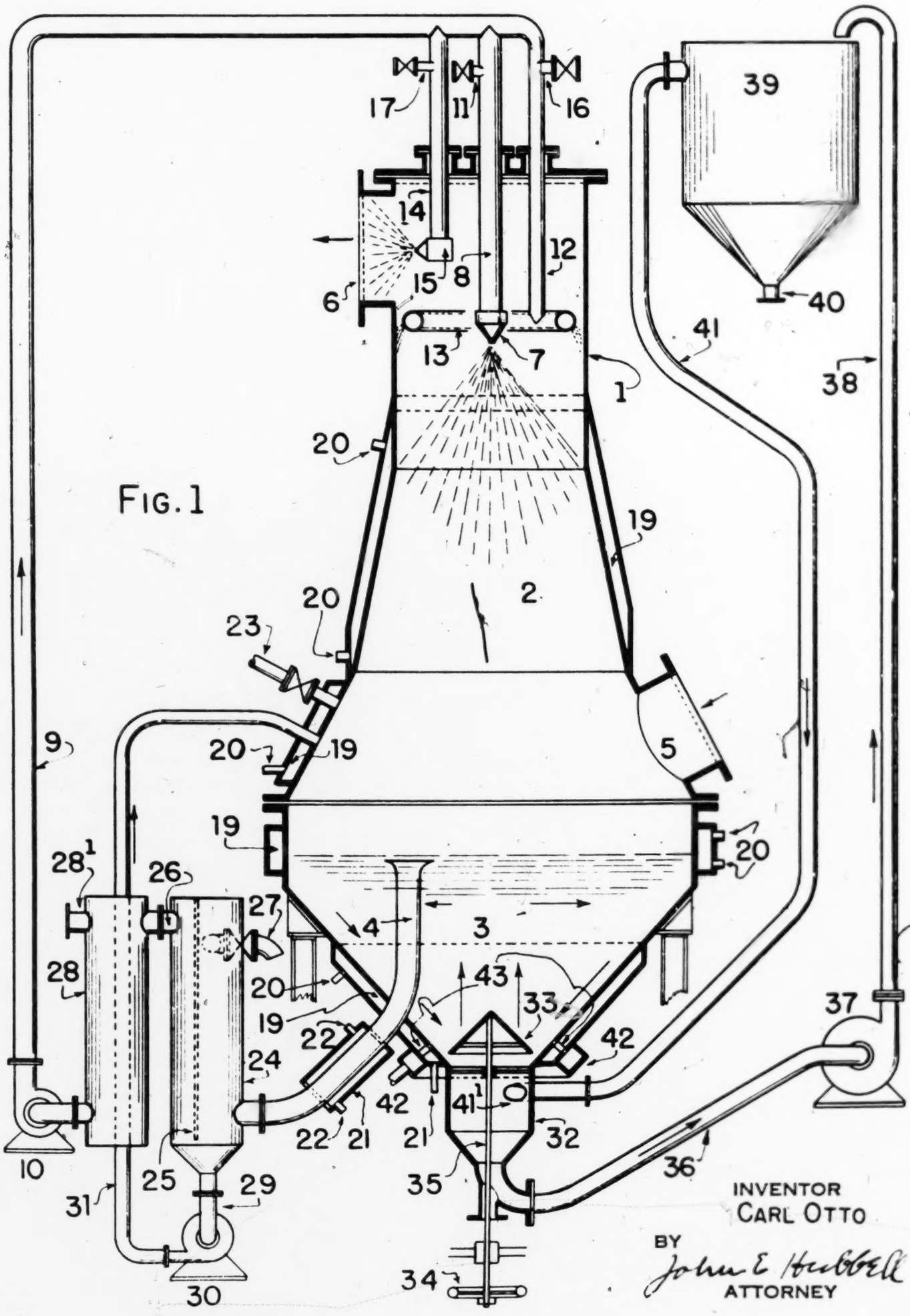


FIG. 1

INVENTOR
CARL OTTO

BY John E Hubbell
ATTORNEY

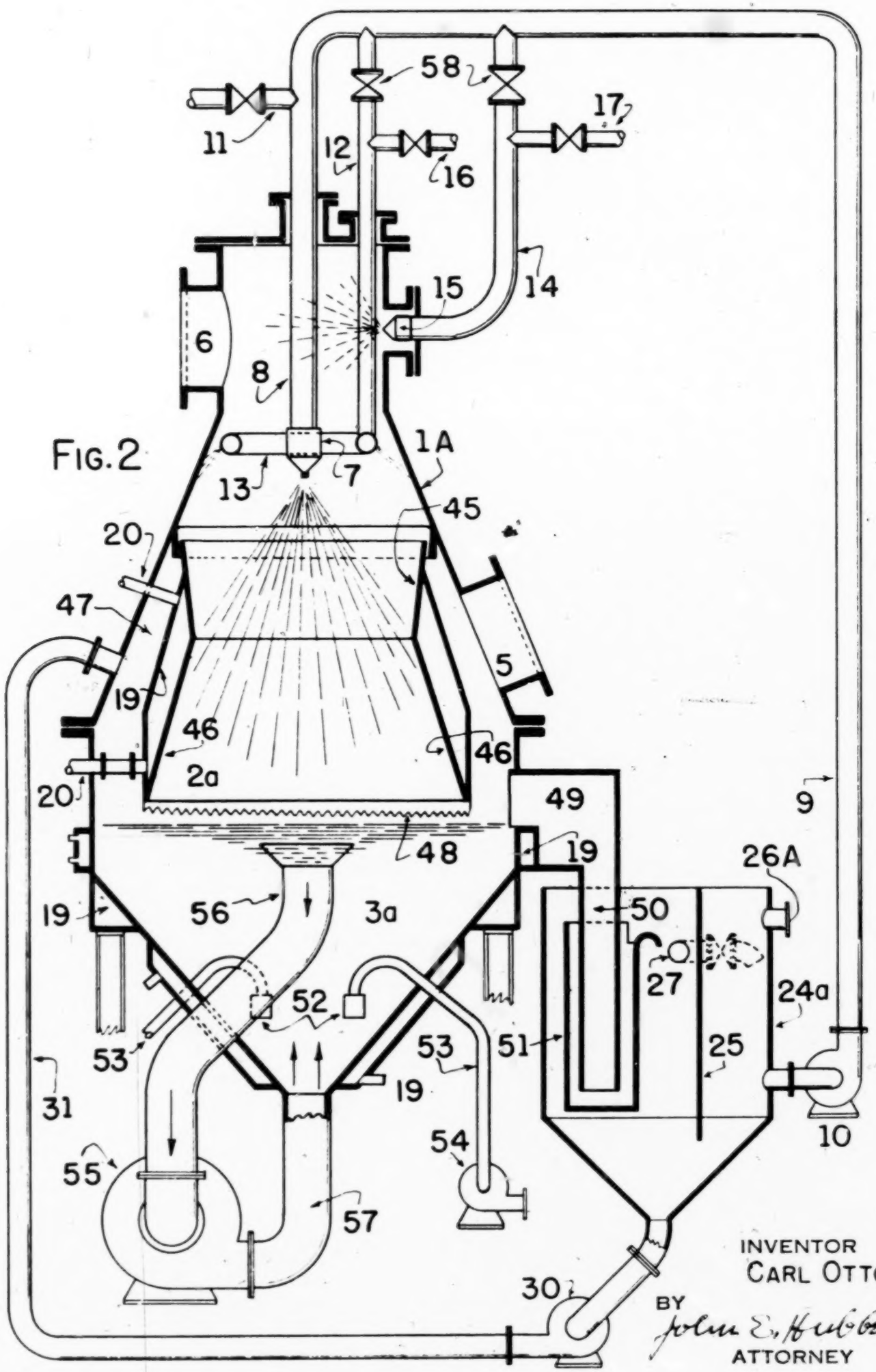
3,1-2

June 3, 1952 C. OTTO 2,599,067

PRODUCTION OF AMMONIUM SULFATE

Filed March 15, 1948 5 Sheets-Sheet 2

FIG. 2



INVENTOR
CARL OTTO

BY John E. Hubbell
ATTORNEY

369-3

June 3, 1952 C. OTTO 2,599,067

PRODUCTION OF AMMONIUM SULFATE

Filed March 15, 1948 5 Sheets-Sheet 3

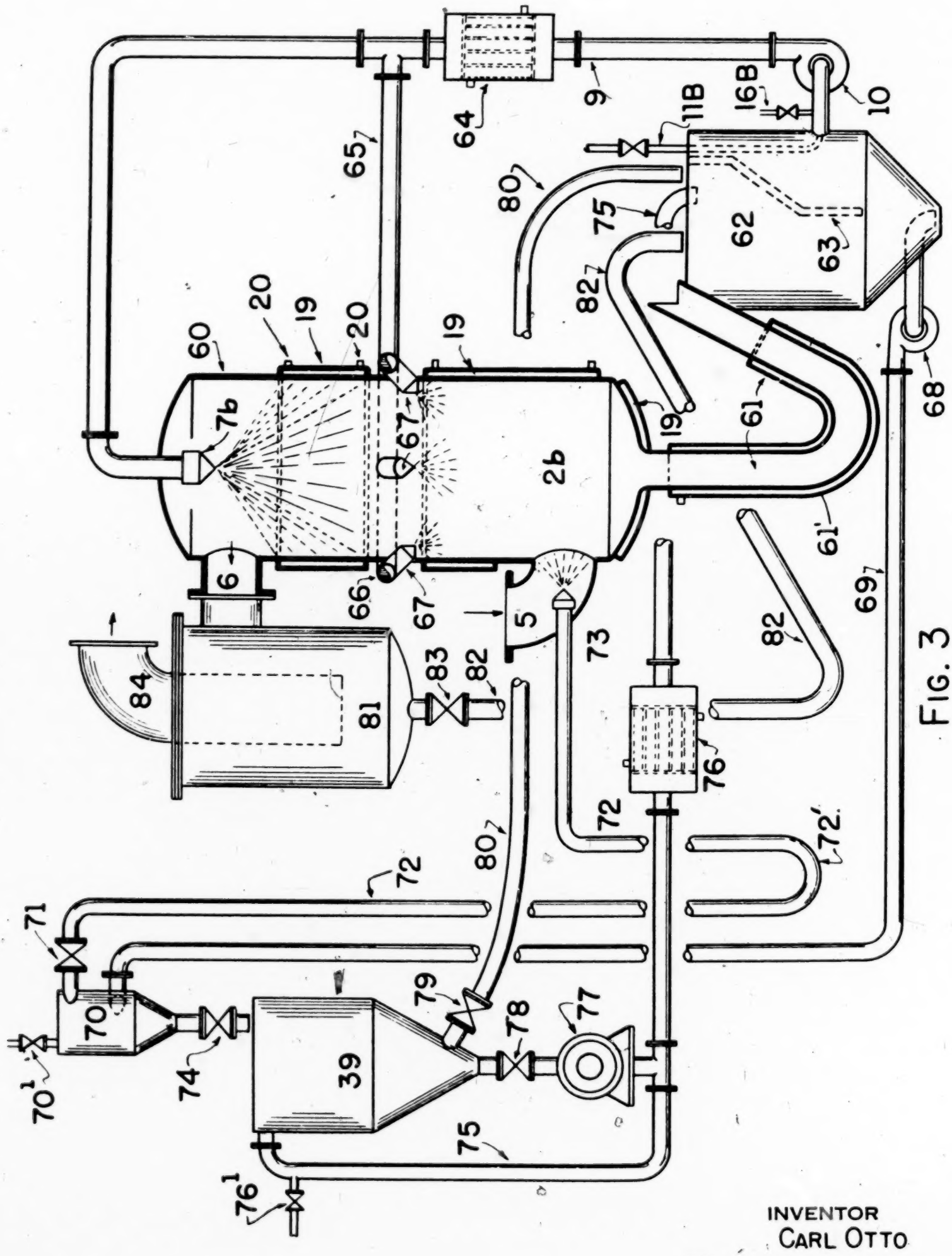


FIG. 3

INVENTOR
CARL OTTO

BY John E. Hubbell
ATTORNEY

June 3, 1952 C. OTTO 2,599,067

PRODUCTION OF AMMONIUM SULFATE

Filed March 15, 1948 5 Sheets-Sheet 4

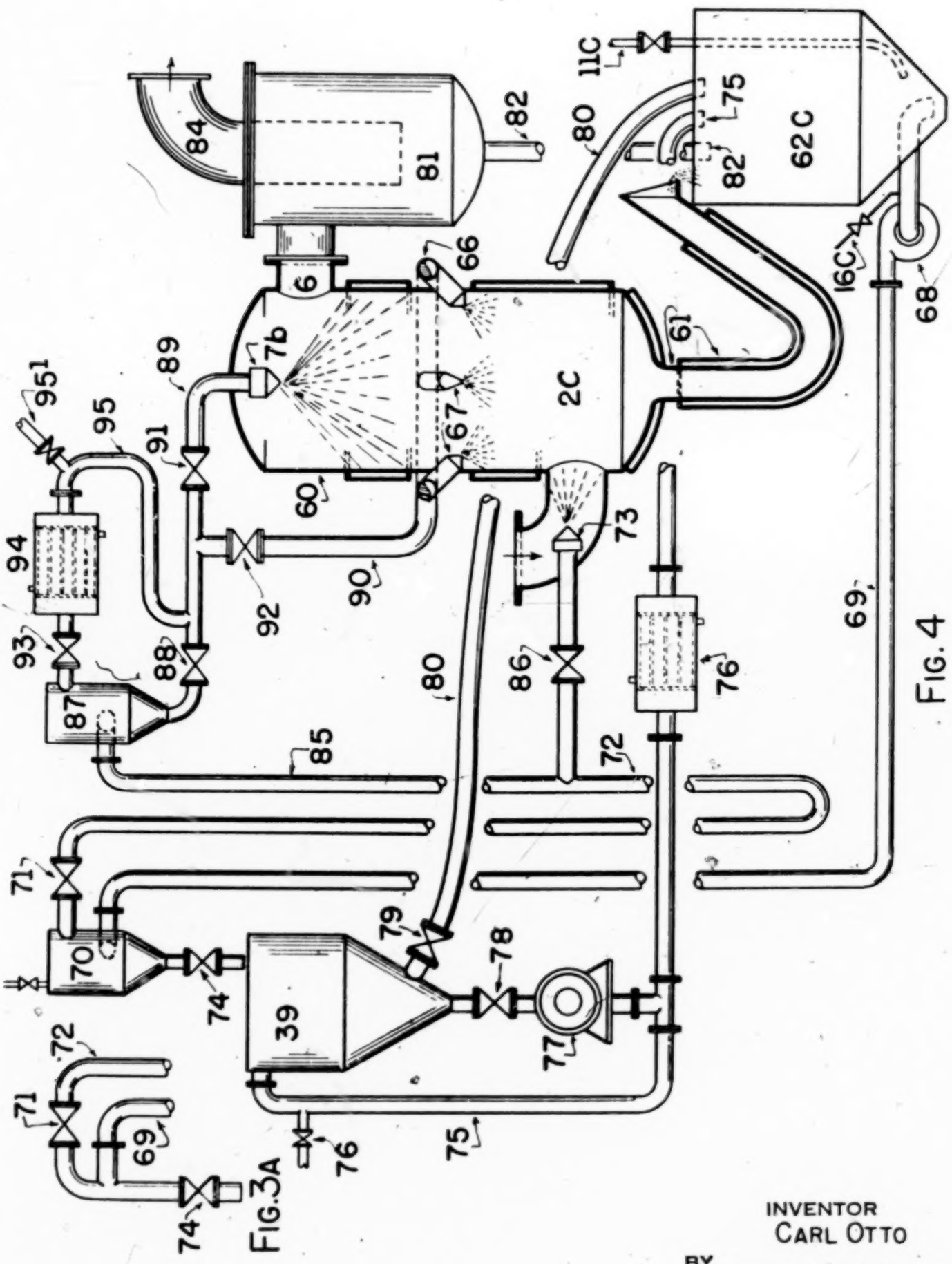


FIG. 4

FIG. 3A

INVENTOR
CARL OTTO

BY John E. Hubbell
ATTORNEY

369-5

June 3, 1952 C. OTTO 2,599,067

PRODUCTION OF AMMONIUM SULFATE

Filed March 15, 1948 5 Sheets-Sheet 5

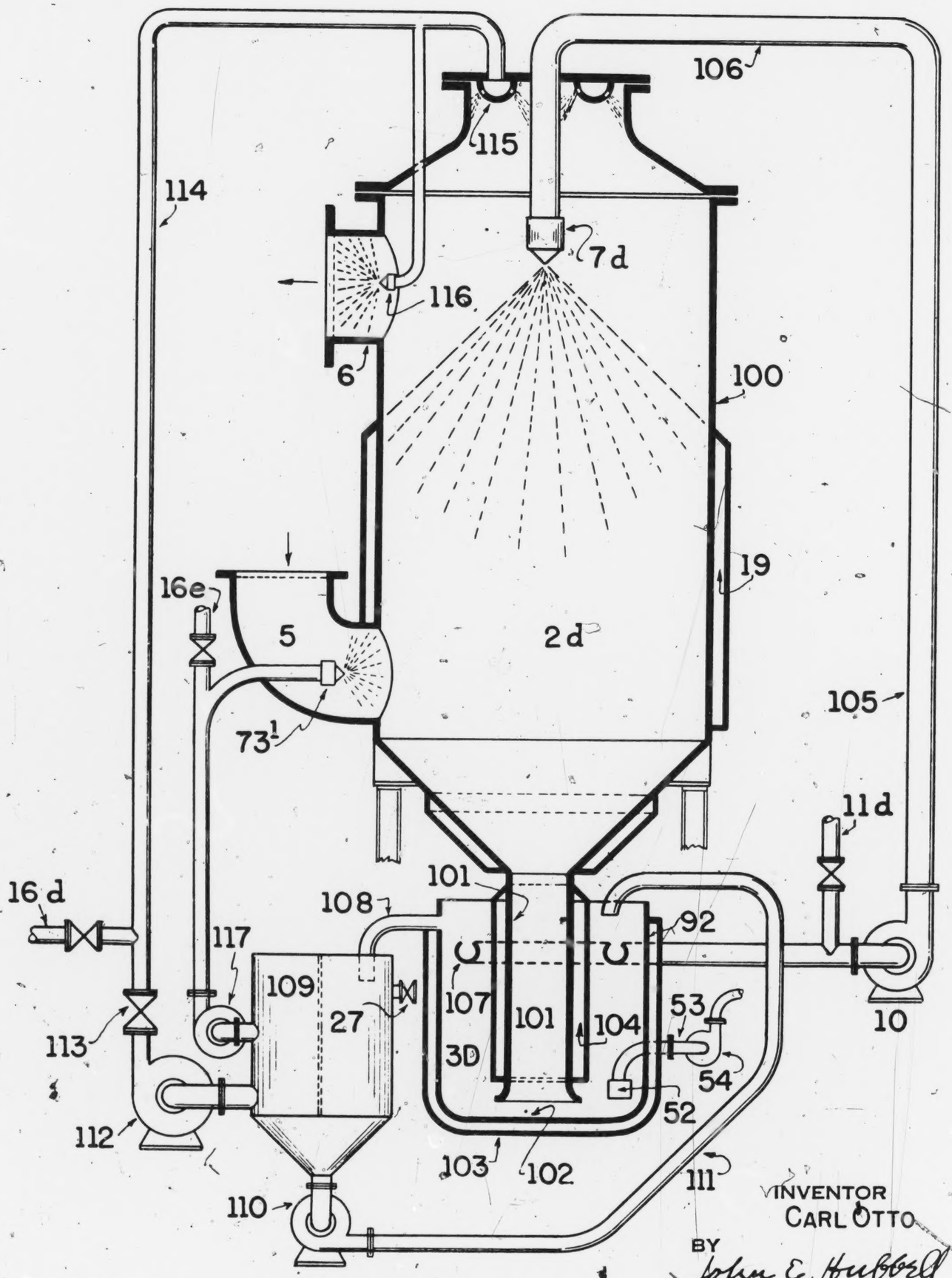


FIG. 5

INVENTOR
CARL OTTO
BY John E. Hubbard
ATTORNEY

Patented June 3, 1952 2,599,067

P. 369-6

UNITED STATES PATENT OFFICE

2,599,067

PRODUCTION OF AMMONIUM SULFATE

Carl Otto, Manhasset, N. Y.

Application March 15, 1948, Serial No. 14,902

In Great Britain December 17, 1947

19 Claims. (Cl. 23—119)

1

The general object of the present invention is to provide an improved method of, and improved apparatus for producing crystalline ammonium sulphate and sulphuric acid to thereby other distillation gas containing ammonia into scrubbing contact with liquor containing ammonia sulphate and sulphuric acid to thereby increase the ammonium sulphate content of the liquor, and thereafter crystallizing ammonium sulphate from the liquor.

A major object of the invention is to provide a method and apparatus for producing sulphate of ammonia which are substantially free from practically important disadvantageous characteristics of the methods and apparatus heretofore commercially used in this country in converting the ammonia content of coke oven gas into sulphate of ammonia.

In converting ammonia in coke oven gas into ammonium sulphate by the method in most general use in this country, now and in the past, the gas is injected into a body of bath liquor containing saturated ammonium sulphate solution and sulphuric acid which is held in the lower portion of a saturator of the "cracker pipe," or submerged gas inlet orifice type. A saturator of the "cracker pipe" type comprises a gas space above the liquor bath and into which the gas bubbles up out of the liquor bath into which it has been injected, and necessarily operates with a gas pressure drop. A desirable form of that type of saturator, having greater capacity per unit of bulk, and operating with a smaller gas pressure drop than prior "cracker pipe" type saturators, is shown in my prior Patent 2,424,206 of July 15, 1947.

The use of a saturator of the "cracker pipe" type in the production of ammonium sulphate, has practically important disadvantages. For one thing, it is practically essential that even the most efficient saturator of the "cracker pipe" type should be of relatively large bulk, and inherently expensive to construct, maintain and operate. Furthermore, the gas pressure drop in such a saturator under normal operating conditions must be large enough to significantly increase the required capacity and operating cost of the machinery employed to move the gas through the saturator. Moreover, in the operation of a saturator of the "cracker pipe" type, solid ammonium sulphate deposits on and adheres to the "cracker pipe" and the internal surface of the saturator wall. The elimination of such deposits, commonly called rock salt, is troublesome and expensive and customarily re-

2

quires a periodical bath killing operation. Each bath killing operation involves the replacement or substantial dilution of all or a substantial portion of the bath liquor in the saturator, by water which dissolves the rock salt.

Aside from the above-described method of using the "cracker type" saturator, the only prior method of converting the ammonia content of coke oven gas into ammonium sulphate in commercial use in this country, is a method described by W. Tiddy in the periodical "Blast Furnace and Steel Plant" for December 1941. In that method, the ammonia is scrubbed out of the gas in a scrubbing column containing a stack of grids, usually tiles, over and through the spaces in which liquor containing unsaturated ammonium sulphate and free sulphuric acid components, flows downwardly while the gas scrubbed flows upwardly. The amount of ammonia absorbed by the stream of liquor flowing down past the grids must be kept too small to supersaturate the ammonium sulphate solution in said streams as such supersaturation would result in rock salt deposits in the grid stack, clogging the latter and increasing the gas pressure drop in the scrubber.

The bath liquor which flows away from the grid stack, is passed from the lower end of the scrubbing column to crystallizing apparatus comprising a crystallizing tank and an elevated evaporator operating under a vacuum. Liquor is pumped into the evaporator from the crystallizing tank. Vaporization in the evaporator eliminates some water from the liquor and lowers the liquor temperature and thereby supersaturates the unevaporated portion of the liquor. Supersaturated liquor is returned through a barometric column connection to the crystallizing tank in which it is desupersaturated as a result of the formation and growth of ammonium sulphate crystals in said tank. The gas pressure drop in the scrubbing column used in the second method may be substantially lower than the pressure drop in the "cracker pipe" saturator, but that advantage is attended by some disadvantages including, in particular, the need for the use of a vacuum evaporator.

In accordance with the present invention, I first produce supersaturated ammonium sulphate by spraying finely divided liquor containing sulphuric acid and ammonium sulphate into a substantially unobstructed scrubbing space or chamber through which a stream of coke oven gas containing ammonia is flowing, whereby the ammonia and acid react to form the additional

ammonium sulphate to supersaturate the ammonium sulphate liquor sprayed into said space. Ordinarily, the ammonium sulphate liquor sprayed into the scrubbing space is saturated, though it is theoretically possible to have the entering spray liquor unsaturated or supersaturated. The spray liquor supersaturated in the scrubbing space or having its degree of supersaturation increased in said space, is then collected in a crystallizing space without necessary change in the temperature or pressure of the liquor. My method thus eliminates the need for using the characteristic "cracker pipe" saturator of the prior method first described, and eliminates the need for using the grid stack and vacuum evaporator used in the second mentioned prior method.

The gas scrubbing space and the liquid crystallizing space used in accordance with the present invention may be respectively formed by the upper and lower portions of a single tank through which the gas passes upwardly and the spray liquor passes downwardly. Advantageously, however, the crystallization is effected in a tank or other receptacle which is separate from the scrubbing chamber tank and which also serves as a tar separator. In an especially desirable form of the invention, the crystallizer comprises a liquid seal between the scrubbing and crystallizing chambers. The crystallizing chamber which is separate from the scrubbing chamber and also comprises a pipe or conduit conveying liquor to the crystallizing chamber from the scrubbing chamber, may also serve as a tar separator. In the crystallizer, the liquor changes from a supersaturated condition into a normal saturated condition.

In the practice of the present invention, ammonium sulphate crystals are formed, and crystals grow in the small liquor drops or particles sprayed into and moving through the scrubbing chamber as the sulphuric acid in said drops or particles unites with ammonia in the gas contacted and thereby supersaturate the liquor in said drops or particles. In all of the forms of the invention disclosed herein, ammonium sulphate crystals are entrained in the liquor sprayed into the scrubbing chamber. In the preferred forms of the invention illustrated in Figs. 3, 3A and 4, at least some of the liquor sprayed into the scrubbing space entrains crystals withdrawn from the portion of the crystallizer in which the larger crystals accumulate. The crystal entraining liquor sprayed into the scrubbing space reacts therein with the ammonia content of the coke oven gas to form ammonium sulphate and thereby supersaturates itself while simultaneously subjecting itself to a desupersaturating action in which ammonium sulphate in the liquor is deposited on the entrained crystals, which are thus caused to grow as they move through the scrubbing space. The major portion of the crystal growth effected and some of the initial formation of crystals effected in the normal use of the invention, occurs in the crystallizing space. The evaporation of water from the ammonium sulphate liquor which occurs in the scrubbing and crystallizing spaces, increases the supersaturation of the liquor from which the water vapor separates. The crystal growth and formation which results from the supersaturation of ammonium sulphate, reduces the amount of ammonium sulphate in solution and thus desupersaturates the ammonium sulphate solution.

In the preferred form of the invention, crystallization is expedited and augmented by recirculation of the spray liquor in the crystallizer space. The ammonium sulphate containing liquor sprayed into the scrubbing space ordinarily consists wholly or mainly of liquor passed more or less directly to the spraying means from the crystallizer. Ammonium sulphate liquor is thus moved along a flow path which is continuous, or closed on itself, and includes the means for spraying the liquor into the scrubber, the scrubbing space, the crystallizing space and conduit connections and pumping means for passing liquor from the last mentioned space to said spraying means. Sulphuric acid must be added to the liquor at some convenient point or points along said flow path and ammonium sulphate crystals must be separated from the liquor. The liquor pumping apparatus required to spray the liquor into the scrubbing space, may serve to produce all, or a major portion of the recirculation of the bath liquor in the crystallizer space which is needed to effect and regulate the crystallizing action.

A further specific object of my invention is to provide a simple and effective method of, and means for preventing rock salt deposits on the walls enclosing the scrubbing and crystallizing space. I have found that the last mentioned object may be substantially obtained by suitably heating, suitably washing, or both heating and washing the wall or walls on which rock salt would otherwise deposit. A suitable wall heating effect may be obtained by a steam jacket or jackets, or by electrical heating structure heating the wall to a temperature high enough to prevent ammonium sulphate crystals from becoming attached to the inner wall surface. The wall washing effect may be obtained by flowing water or bath liquor along the wall in a film-like stream of sufficient volume and flow velocity to prevent ammonium sulphate crystals in said stream from adhering to said wall.

In the practice of my method, as in the practice of the two prior methods hereinbefore described, the gas passing away from the apparatus in which the ammonia content of the gas is recovered, entrains bath liquor which is recovered in a so-called acid catcher. Such recovery is practically essential in all cases because the cost of the sulphuric acid used is a major cost item in the process, and also because the inclusion of more than a trace of sulphuric acid in the gas makes the latter undesirably corrosive. In the practice of the present invention I have found that a decrease in the average size of the subdivided liquor particles sprayed into the gas stream increases the absorption efficiency of the sprayed liquor but also tends to increase the amount of the gas entrained liquor carried into the acid catcher. A specific object of the present invention is to make it possible to obtain the advantage of spraying finely sub-divided liquor into the scrubbing space while minimizing the passage of the finely subdivided liquor into the acid catcher.

I have discovered that the last mentioned object of the invention may be obtained by spraying bath liquor which is sub-divided relatively coarsely into the gas as it passes through the portion of the scrubbing chamber, preferably the upper portion, adjacent the gas outlet of said chamber, and simultaneously spraying relatively finely sub-divided liquor into the portion of the scrubbing chamber more remote from the gas exit to the acid catcher. Preferably the amount of finely sub-divided liquor sprayed into the gas

is sufficient to absorb the major portion of the ammonia content of the gas. With this spraying procedure, the relatively coarse liquid particles engage and absorb, or coalesce with finer particles of liquor sprayed into the portion of the scrubbing space more remote from the exit. By the use of a suitably large portion of the spray liquor in the form of a coarse spray, substantially all of the fine liquor particles entrained in the gas stream may be eliminated from said stream. The coarse spray serves to absorb substantially all of the ammonia content of the gas not absorbed by the fine spray.

A plurality of pumps may be used with advantage in some cases to supply acidified liquor to the several spraying devices and to effect the desired recirculation of the liquor required for the desired crystal growth, but it is practically possible to use only a single pump.

In the practical use of the present invention, I have found that good operating results are obtainable with a gas upflow velocity in the scrubber of from 1 to 23 feet per second, and with liquor sprayed into the upper portion of the scrubber adjacent the gas outlet in the form of a coarse spray comprising liquor particles or droplets having an average diameter of more than two millimeters, and with liquor sprayed into a lower portion of the scrubber in the form of a fine spray comprising particles or droplets having an average diameter slightly below 0.5 millimeter. In such operation, most of the fine liquor spray entrained in the gas stream is knocked out of the latter by the coarse spray in the upper portion of the scrubbing space, and the amount of acid carried into the external acid catcher by the outflowing gas is relatively very small. In such practical use of the invention I have found that for the satisfactory removal of the ammonia in the coke oven gas passing through the scrubber, without passing an excessive quantity of spray liquor to the acid catcher and without requiring excessively large scrubber dimensions, the total quantity of liquor sprayed into the scrubber should ordinarly exceed 3000 gallons per short ton of ammonia sulphate produced.

The various features of novelty which characterize my invention are pointed out with particularity in the claims annexed to and forming a part of this specification. For a better understanding of the invention, however, its advantages, and specific objects attained with its use, reference should be had to the accompanying drawing and descriptive matter in which I have illustrated and described preferred embodiments of the invention.

Of the drawings:

Fig. 1 is a somewhat diagrammatic sectional elevation of one form of my improved apparatus for utilizing the ammonia content of coke oven gas in the production of crystalline ammonium sulphate;

Figs. 2, 3, 4 and 5 are views generally similar to Fig. 1, and each illustrating a different modification of the apparatus shown in Fig 1; and

Fig. 3A is an elevation illustrating a modification of parts of the apparatus shown in Fig. 3.

The apparatus shown in Fig. 1 comprises a vertically disposed tank or casing structure 1 enclosing an upper gas scrubbing space or chamber 2, and a lower liquid holding crystallizing space or chamber 3. The gas and liquid holding spaces or chambers 2 and 3 of Fig. 1 are in open communication with one another at the level at which liquid overflows from the space 3

into the overflow outlet pipe 4. The tank 1 is preferably circular in cross-section and may well be of uniform diameter from top to bottom. As shown, however, the lower liquid holding portion of the tank is of conical or hopper bottom shape, and the gas space 2 is tapered so that it is larger in horizonal cross-section at its lower end than at its upper end. The gas to be scrubbed enters the lower portion of the gas space 2 through an inlet 5 and passes away from the upper end of the space through an outlet 6.

In accordance with the present invention, acidified ammonium sulphate liquor is sprayed into the scrubbing chamber 2 at the upper end of the latter through a central, downwardly directed, spray nozzle 7. The latter is connected to the lower end of a vertically disposed pipe 8 which enters the space 2 through the top wall of the latter, and which forms a branch of the outlet pipe 9 of a pump 10 normally discharging saturated ammonium sulphate liquor under pressure. As shown, sulphuric acid may be added to the liquor passing to the nozzle 7 through the pipe 8, by a valved acid supply pipe 11. The pipe 9 also supplies ammonium sulphate liquor through a branch pipe 12 to a horizontal spray ring 13 in, and adjacent the upper end of the scrubbing chamber 2. The spray ring 13 is formed with a multiplicity of discharge orifices distributed about the axis of the tank 1 and directed downwardly and outwardly so as to maintain a film-like stream of liquid flowing downward along the peripheral wall of the chamber 2. Through a third branch 14, the pipe 9 supplies liquor to one or more spray nozzles 15 provided near the gas outlet 6. The spraying devices 13 and 15 flush with bath liquor the inside walls of the gas space 2 and gas outlet 6. The liquid discharged by the spray nozzles 13 and 15 minimizes rock salt deposits on the wall surfaces flushed or washed by said liquid.

While in normal operation, the flushing liquid discharged through the nozzles 13 and 15 may be saturated ammonium sulphate liquor, the branch supply pipes 12 and 14 are shown as provided with valved pipe connections 16 and 17 through which hot water or hot water admixed with more or less saturated ammonium sulphate liquor, may be supplied to the nozzles 13 and 15, respectively, when necessary to eliminate rock salt deposits in the saturator. Further protection against rock salt deposits, is provided by the wall heating jackets 19 surrounding different portions of the tank structures, with pipe connections 20 for the passage of steam into, and the discharge of water of condensation from the different heating jackets. The temperature of the wall portions surrounded by the jackets 19 may be kept high enough to prevent rock salt deposits on the internal surfaces of the wall portions respectively adjacent the different steam jackets.

As shown, an external portion of the overflow pipe 4 is surrounded by a steam jacket 21, having pipe connections 22. A valved pipe connection 23, shown as opening into the scrubbing chamber 2 adjacent its lower end, forms a second means through which sulphuric acid may be added to the liquor in the subjacent crystallizing chamber. The pipe connection 23 forms an especially appropriate means for making use of the reclaimed acid from a benzol recovery plant (not shown) through which the coke oven gas is passed subsequent to its passage through the scrubbing chamber 2.

The overflow pipe 4 passes the overflowing

liquor into the lower portion of a seal pot receptacle 24 at one side of a baffle 25 which does not extend to the bottom of the receptacle, but does prevent tar entering the receptacle 24 through the pipe 4, from passing out of the receptacle through the overflow pipe 26 at the opposite side of the baffle 25. The receptacle 24 serves as a tar separator of well known type, and a valved pipe connection 27 to the portion of the receptacle 24 at the right of the baffle 25 forms a means through which tar collecting in the upper portion of said receptacle portion may be periodically discharged. The overflow outlet 26 from the clear liquor chamber of the receptacle 25 at the left of the baffle 25, opens into the upper end of an overflow receptacle 28. The latter serves as a liquor supply chamber for the pump 10. The latter has its inlet connected to the lower end of the overflow receptacle. As shown, the receptacle 28 is provided with overflow outlet 28' through which overflowing liquor may be passed into a conventional mother liquor reservoir (not shown). The tar separator 25 has a bottom outlet 29 connected to the inlet of a liquor circulating pump 30. The latter, as shown has a discharge pipe 31 opening into the lower portion of the scrubbing chamber 2.

The conical, liquor containing, crystallizing chamber 3 opens at its lower end into a subjacent crystal receiving chamber 32. The latter is in the form of a vertically disposed cylinder and has an open upper end which is in communication with the lower end of the conical crystallizing chamber 3 to an extent which may be variably restricted by the adjustment of a valve 33. The valve 33 may be vertically adjusted by means of a hand wheel 34 secured to the lower end of the spindle 35 of the valve 33. The crystal chamber 32 has a bottom outlet connected by a pipe 36 to the inlet of a slurry pump 37 discharging through its outlet pipe 38 into an elevated crystal settling tank 39. The latter is provided with a bottom outlet 40 through which crystals may be discharged to a crystal drying filter or centrifuge device such as the device 77 shown in Fig. 3. Liquor passes away from the upper portion of the settling chamber 39 through an overflow pipe 41 and is returned to the chamber 32 through a tangential inlet 41'. Communication between the crystal receiving chamber 32 and the crystallizing chamber 3 may be cut off or throttled by the valve 33 as conditions make desirable.

To effect or augment recirculation and thereby further the crystallizing action in the chamber 3, air, gas or liquor may be injected into the body of liquor in the lower portion of the chamber 3 through the distributed outlet nozzles 43 from a spray ring 42. The jets discharged through the nozzles 43 also reduce the amount of relatively small crystals entering the chamber 32.

In the normal contemplated operation of the apparatus shown in Fig. 1, coke oven gas is continuously passed into the scrubbing chamber 2 through the lower inlet 5, and passes away from the chamber 2 through the upper outlet 6. As the gas passes upward through the scrubbing chamber 2 and thence through the outlet 6, it is subjected to the scrubbing action of the bath liquor sprayed into said chamber. The major portion of the scrubbing and wall washing liquor needed, is supplied to the scrubbing chamber through the nozzle 7, and is so sub-divided and directed as to be dispersed throughout the entire cross-section of the portion of the scrubbing chamber 2 between spaced apart upper and lower levels, respectively below the nozzle 7 and above the gas inlet 5. Furthermore, the amount of liquid discharged by the nozzle 7 needs to be sufficient to absorb all of the free ammonia carried into the scrubbing chamber by the gas, except the relatively small portion of said ammonia which is absorbed by the liquor supplied by the spraying devices 13 and 15, most of which passes downward to the crystallizing space 2 in the form of a down-flowing film on the tank wall. Substantially all of the free ammonia is normally removed from the gas before the latter passes into the space into which liquor is sprayed by the nozzle or nozzles 15. The primary purpose of the spray nozzles 15 is to flush the inside surface of the upper portion of the scrubber shell and gas exit pipe.

The spray liquor passing down through the scrubbing chamber 2 is supersaturated when it reaches and passes into the body of liquor in the crystallizing space 3. The liquor film flowing down the portion of the tank wall surrounding the scrubbing chamber 2 and absorbing ammonia from the gas coming into contact therewith, has its ammonium sulphate content increased by spray liquor discharged by the nozzle 7 and passing into said film after passing through enough of the gas space to become supersaturated. While the liquor forming the film flowing down the wall normally consists of supersaturated ammonium sulphate, it has but little tendency to form rock salt deposits on the wall of the chamber 2, if the volume of the film liquor is sufficient to provide an adequate flushing or wall washing action. When such deposits occur, or tend to occur, hot water may be added through the connections 16 and 17, to the liquor passing through the nozzles 13 and 15 as required to prevent or remove such deposits. Moreover, the formation of rock salt deposits on the walls surrounding the chambers 2 and 3 may be substantially prevented by the heating action of the steam jackets 19 which may be used to heat the film of ammonium sulphate in immediate contact with the tank wall so as to thereby reduce the supersaturation of the film liquor sufficiently to prevent rock salt deposits on the tank wall.

While the liquor passing into the liquid filled crystallizing space 3 from the scrubbing chamber 2 may be highly supersaturated, it is quickly desupersaturated as ammonium sulphate crystallizes out of solution to form seed crystals or to become part of the crystal growth which occurs in the crystallizing chamber and increases the size of the crystals therein. The growth of the crystals is expedited and augmented by the recirculation of the liquor in the crystallizing chamber due to the action of pumps 10 and 30. All, or the major portion of the liquor drawn from the crystallizing chamber by each of those pumps is returned to that chamber through one or more of the spray nozzles 7, 13 and 15, and the pipe 31.

In the crystallizing space, the larger crystals settle more rapidly than do the smaller crystals. In consequence, the crystals passing away from the crystallizing chamber through the overflow pipe 4 are of smaller average size than the crystals withdrawn from the crystallizing chamber through the crystal chamber 32. The liquor returned to the chamber 32 through the pipe 41 carries with it much of the small crystal content passed into the crystal separating tank 39 through the pipe 38. The small crystals in the chamber 32 tend to continuously increase in size. As those skilled in the art will recognize, the average size of the crystals produced may be regulated by varying the conditions of operation of the apparatus disclosed herein. Furthermore, known special expedients may be used to produce large crystals when such crystals are desired.

It is practically desirable that the scrubbing liquor spray should contain saturated rather than unsaturated or supersaturated liquor, and it is essential that in normal operation the liquor which has been sprayed into the scrubbing chamber or space 2, should be in a supersaturated condition when it passes from that chamber into the crystallizing chamber or space. However, a considerable variation in the extent or degree of supersaturation is permissible, and in normal practice it is generally desirable that the supersaturation should not be so high that seed crystals of ammonium sulphate will be formed in the crystallizer bath liquor rapidly enough to interfere with the desired crystal growth.

As those skilled in the art understand, the liquor which I use to scrub free ammonia out of coke oven gas in the production of ammonium sulphate, consists mainly of ammonium sulphate solution along with some sulphuric acid and a small amount of ammonium sulphate in crystal form. The reaction between ammonia and sulphuric acid is exothermic and increases the temperature of the liquor. The coke oven gas coming to the scrubbing chamber 2 carries some water vapor and is at a temperature which normally is within the range of 120° to about 150° F. When the gas coming into the scrubber has a constant temperature and water vapor content, the liquor composition stabilizes with ammonium solution dilution dependent on the said temperature and the water vapor content of the gas. An increase or decrease in the water vapor content of the gas, without a compensating change in the gas temperature, results in a decrease or increase respectively, in the ammonium sulphate content of the liquor, when the sulphate is in its normally saturated condition.

In continuous commercial use, in the general manner above described, of apparatus of the type and form shown in Fig. 1, good operating results are obtained when the average gas temperature in the scrubbing space 2 and liquor temperature in the crystallizing space 3 are each between 120° F. and 150° F., and the liquor in the chamber or space 3 has an average acid content of about 5 or 6 percent, and average density of about 1.3, and above 3000 gallons of liquor is sprayed into the scrubbing space 2 for each 2000 pounds solid ammonium sulphate produced. Neither the precise acid content of the liquor in the crystallizing space 3 or being sprayed into the scrubbing space 2, nor the precise density of the liquor in either space is critical. Ordinarily, it is practically undesirable to have the acid content of the liquor in the crystallizing space or chamber above 6 per cent, since a higher acid content in said space tends to result in an acid content in the finished crystalline ammonium sulphate produced, which, while small, is objectionable, particularly because of its corrosive effect. It is practically essential, of course, that the acidity and volume of the liquor sprayed into the scrubbing space 2 should be high enough to insure the conversion of all the ammonia content of the gas into ammonium sulphate.

The liquor sprayed into the space 2 should be sufficient in amount, and suitably dispersed, to keep a substantial portion, at least, of the vertical extent of the space 2 filled with the liquor spray, and the amount of spray liquor and its initial acid content should be such as to prevent a reduction in the acid content of the spray liquor in the scrubbing space below about 4 percent. Thus, for example, if the spray liquor passing through the nozzle 7 of Fig. 1 has an acid content of 9 percent, the liquor passing into the crystallizing space bath from the scrubbing space 2 should have an average acid content of not less than about 5 percent.

The amount of spray liquor used should be sufficient to prevent the sulphate of ammonia content of the spray liquor from rising more than about 5.4 percent above the saturation content, as otherwise the supersaturation of the liquor passing from the scrubbing space to the crystallizing space will become high enough to cause the formation of an undesirably large number of small crystals, with an undesirable resultant reduction of the capacity of the supersaturated liquor passing into the crystallizing space 3 to produce a suitably great crystal growth in said space. The differences mentioned in the immediately preceding paragraphs between the acid content of the liquor sprayed into the scrubbing chamber, and the acid content of the supersaturated ammonium sulphate passing from the scrubbing space into the crystallizer space, are differences existing when the amount of spray liquor used in the production of a ton of ammonium sulphate approaches the minimum, and are decreased when the amount of spray liquor used is increased to increase the crystal growth and the size of the crystals produced. The progressive increase in horizontal cross-section of the scrubbing space as the distance below the spray device 7 increases, coupled with the expanding cross-section of the liquor spray as the distance below the nozzle 7 increases, has the theoretical but practically unimportant effect of permitting a substantial portion of the vertical extent of the scrubbing space to be kept filled with the spray discharged by the nozzle 7, without causing much of the spray to impinge against the wall surrounding the scrubbing space.

In the normal use of the invention, the acidity of the bath liquor is advantageously measured in any usual or suitable manner, continuously or at frequent intervals, and the rate at which acid is added to ammonium sulphate liquor in the tank 1 through the valved pipe connections 11 and 23 or otherwise, is increased or decreased in accordance with the measurements. In general, it is practically feasible to supply make-up acid to the liquor circulating system at any convenient point. With apparatus of the type shown in Fig. 1, the acid may be added in whole or in part through the pipe 11 to the scrubbing liquor about to be sprayed into the scrubber, or through the pipe 23 to the portion of the scrubber space immediately above the crystallizer bath space, or the acid may be passed into the crystallizing bath at various points as is illustrated in Figs. 3 and 4, or into inlet of the pump 10 as is shown in Fig. 5.

The supersaturated liquor passing from the scrubbing space 2 into the crystallizing space 3 is desupersaturated in the last mentioned space as a result of the loss of ammonium sulphate from

P 357 (1)

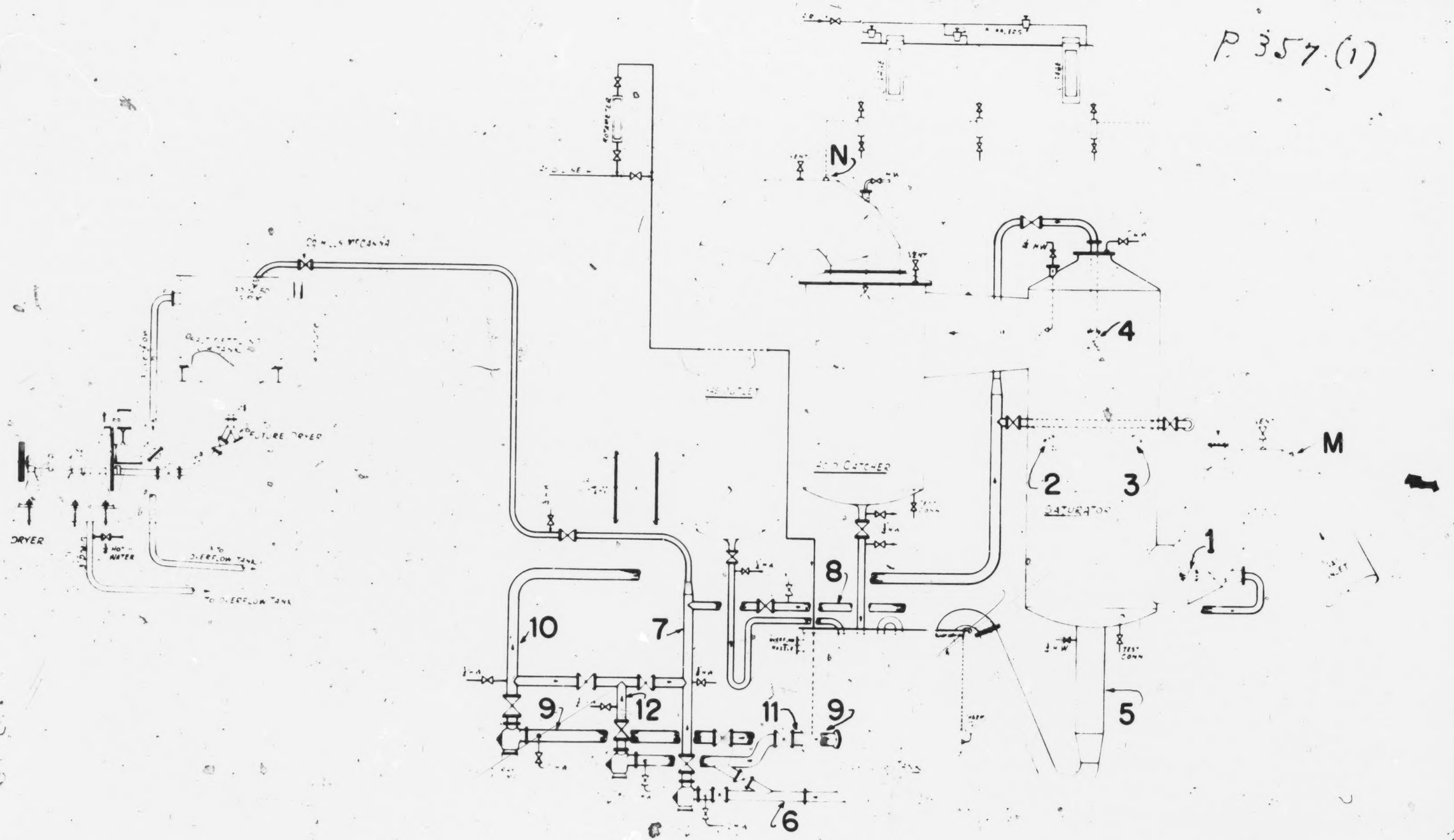


EXHIBIT C
DRAWING

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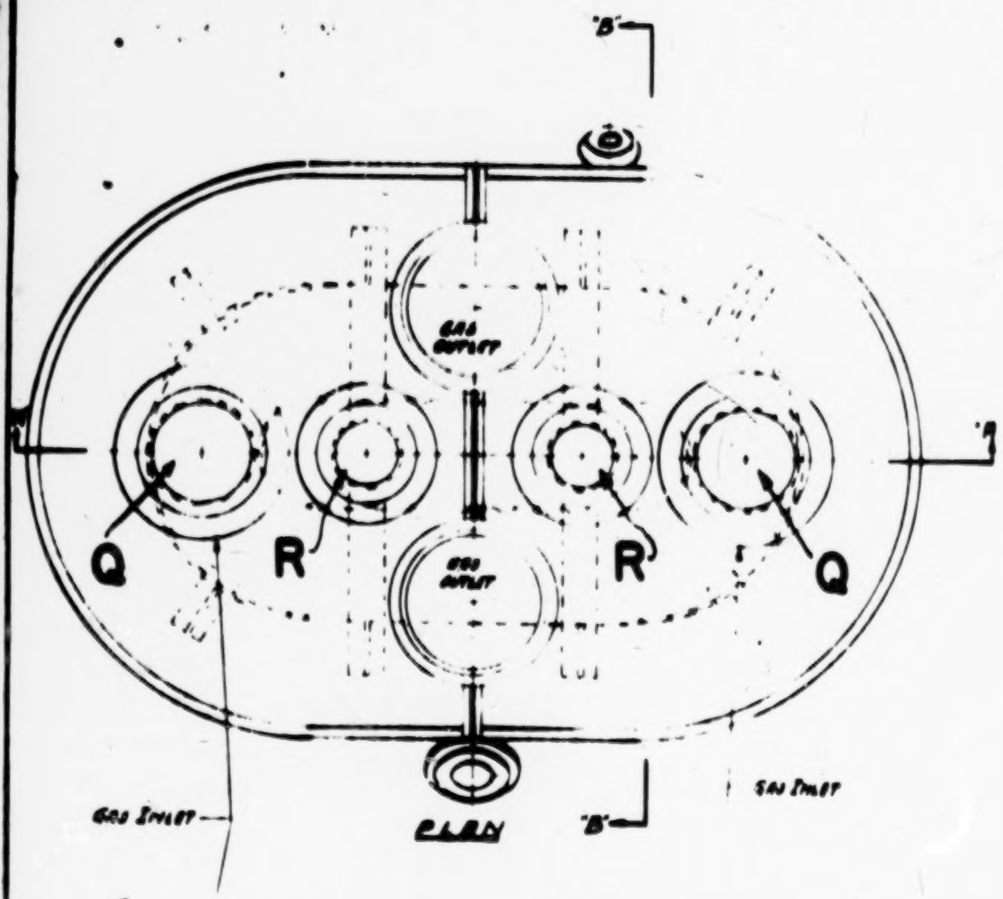
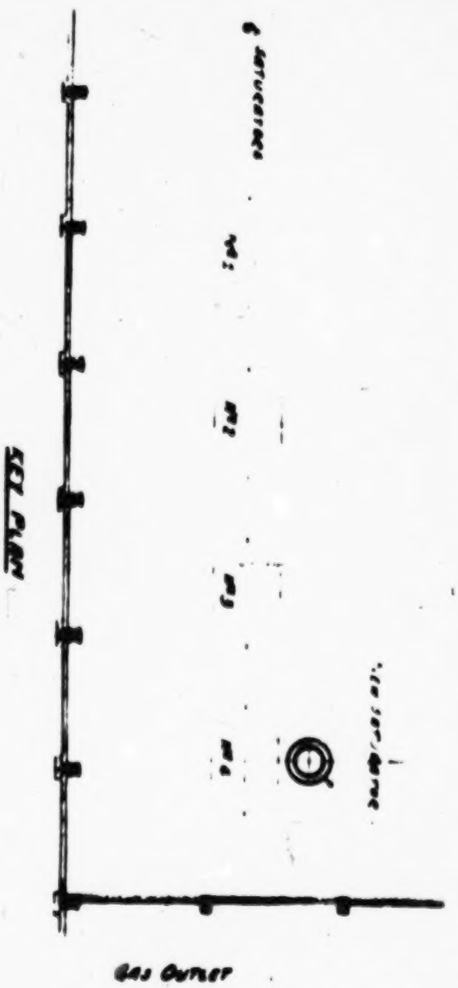
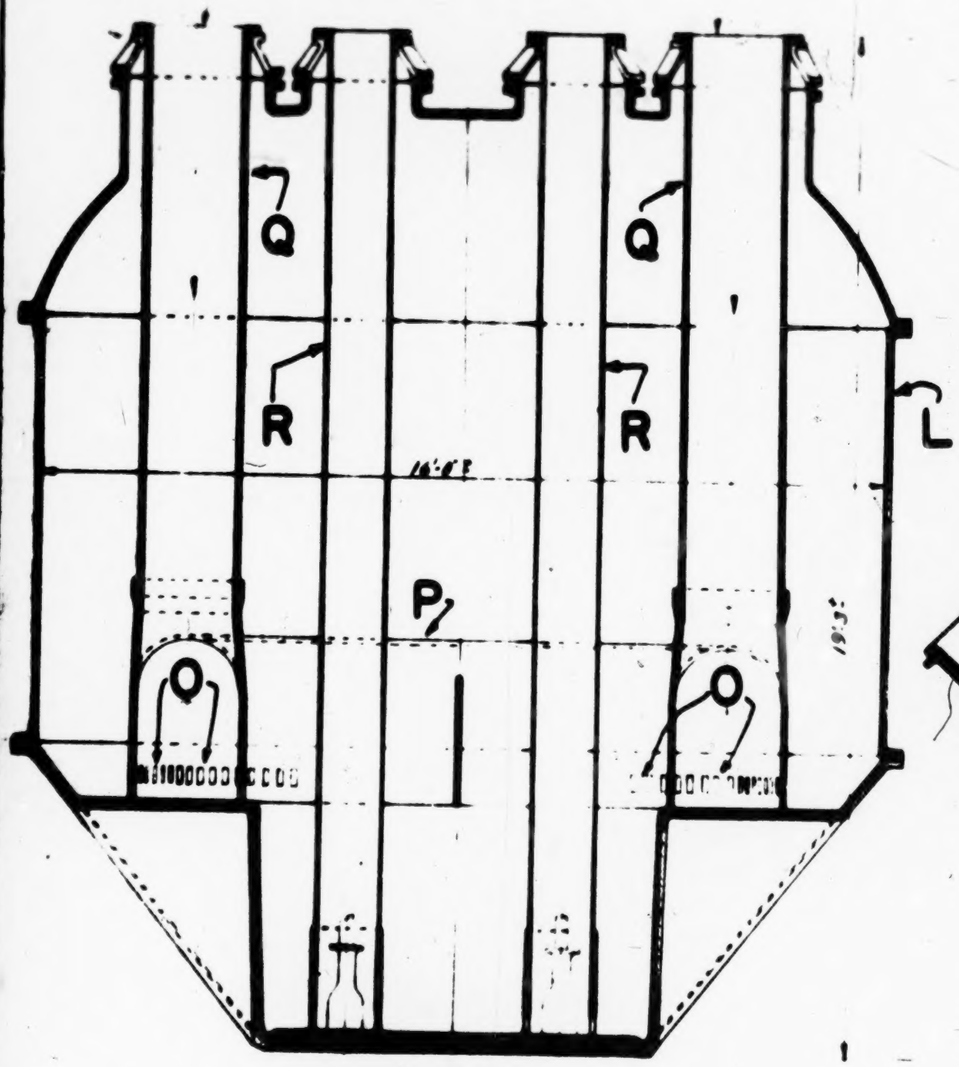
	Bingham Steel Co Franklin Pierce Borough	660 2479

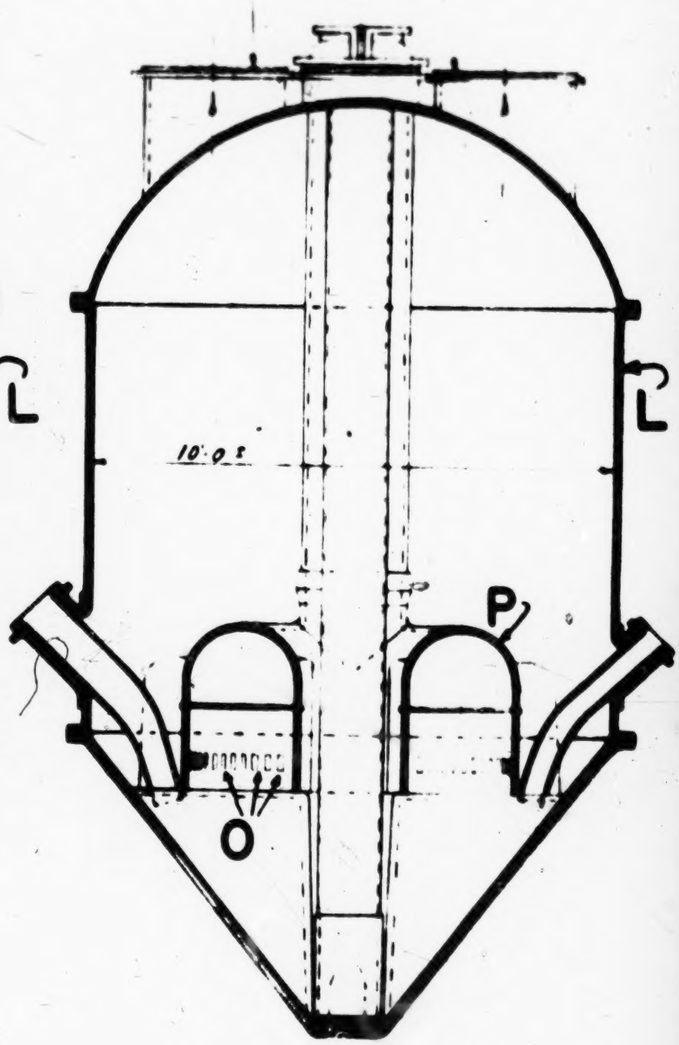
P. 357 (2)







SECTION "A-A"



SECTION B-B

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EXHIBIT D
DRAWING

the liquor to the crystals growing and formed in the chamber 3. In consequence of the crystal growth and the resultant desupersaturation of the liquor, saturated liquor overflowing from the tar separator normally contains a relatively small amount of small crystals and practically no large crystals, since the latter settle out in the crystallizer spaces.

The removal of tar in such manner as to keep the amount of tar in the crystallizing space relatively small, favors the production of ammonium sulphate having a good white appearance. When necessary to facilitate a desirably rapid removal of tar from the tank 24 through the valve discharge outlet 27, liquor may be passed into the tank 24 through the overflow tank 28 from the mother liquor reservoir (not shown). By keeping the crystals in suspension in the crystallizing space and removing them from the bath at a controlled rate, the size of the crystals produced can be controlled by the operator to meet the requirements of the trade. In this way, large crystals can be produced, for example, crystals of an average size such that 40 percent thereof are too large to pass through a 35 mesh screen, and 5 percent or less will pass through a 70 mesh screen. The return of liquor from the lower end of the tar separator 24 to the tank 1 by the pump 30, not only desirably augments the recirculation in the crystallizer space 3 and effects the removal of the tar from the space 3, but contributes to the production of large crystals by desirably minimizing the number of small crystals sprayed into the scrubbing space through the nozzles 7, 13 and 15. The crystal growth in the crystallizer space 3 is relatively rapid, particularly as a result of the agitation or relative movement of the liquor and crystals which increases the extent of contact of the crystals with supersaturated liquor. The liquor in the space 3 of Fig. 1 is agitated and given motion as a result of the operation of each of the pumps 10 and 30, and the liquor agitation may be further increased by air, gas or liquor injected into the chamber 3 through the nozzles 43.

Apparatus constructed in accordance with the present invention is characterized by its relatively small bulk relative to output. Thus, for example, I have found that it is practically feasible to pass coke oven gas at the rate of 80 million cubic feet per 24 hour day through a tank of the general form and proportions of the tank 1 of Fig. 1, with an internal diameter of 11 feet at the top of the bath and of 8 feet at the level of the gas outlet 6. No prior apparatus for the recovery of ammonia from coke oven gas of any type known to me has as great a capacity per unit of bulk. In consequence of its relatively small bulk, it is readily feasible from the practical economic standpoint to make all of the apparatus exposed to contact with the bath liquor, of an alloy, preferably Monel metal, which is immune to corrosive attack by the bath liquor with its substantial sulphuric acid content.

The operating efficiency of the apparatus shown in Fig. 1, may, and ordinarily will be augmented by returning to the tank 1 the liquor separated from the gas in the acid catcher through which the gas is passed from the scrubbing space outlet 6, and by returning to the tank 1 the liquor separated from the crystals passing from the crystal settling tank 39 through its outlet 40 to the crystal drying apparatus. No acid catcher or crystal drying devices are shown in Fig. 1, but they may be of any usual or suitable type, and may be arranged as are the acid catcher 81 and the crystal dryer 77 shown in Fig. 3.

As has been previously stated, the general principles and advantages of the invention may be utilized and realized with apparatus quite different in form from that shown in Fig. 1. Examples of some such other forms of apparatus are illustrated in some detail in Figs. 2, 3, 4 and 5. Each of those figures includes some elements and characteristics similar in function and purpose to, and not significantly different in form from corresponding elements and characteristics shown in Fig. 1, and are designated by the same or analogous reference symbols. Thus, for example, the analogous symbols 2, 2*a*, 2*b*, 2C and 2*d* are used to designate the scrubbing space in Figs. 1, 2, 3, 4 and 5 respectively.

While the apparatus unit shown diagrammatically in Fig. 2 is quite similar to the apparatus shown in Fig. 1 in many respects, it differs from the Fig. 1 apparatus in several material respects. For one thing, the scrubbing chamber or space 2*a* of Fig. 2 is surrounded by a wall including two hollow members 45 and 46, each of which is in the form of a truncated cone. Each of said members is spaced inwardly from the tank or shell 1*a* which encloses the scrubbing space 2*a* and crystallizing space 3*a* of Fig. 2. The elements 45 and 46 are coaxial but are tapered in the opposite direction, the base of the conical member 45 being at its upper end and the base of the conical member 46 being at its lower end. The diameter of the lower end of the member 45 is equal to the upper end diameter of the member 46. The latter is so disposed and proportioned relative to the location of the nozzle 7, and relative to the conical angle of the spray discharge by said nozzle that said spray is distributed substantially across the full cross-section of, and fills the member 46 so that most of said spray passes downward through the member 46 without contacting the latter. The members 45 and 46 collectively form a hollow walled skirt which includes a wall heating space 19 and which is spaced away from the surrounding, conical portion of the tank or casing 1A to provide, an annular gas receiving space 47. The incoming gas passes into the space 47 through the inlet 5 at a level appreciably above the lower edge 48 of the member 46. That edge is serrated and breaks up the stream of gas which flows from the inlet port 5 downward through the space 47 into the space below the member 48 and above the bath liquor in the chamber 3*a*, and passes from said space upward into and through said space. The liquor flowing in film form down the inner surface of the member 46 is also broken up into drops which fall from the serrated bottom edge 48 of the member 46, and is the portion of the ammonium sulphate liquor with which the gas entering the apparatus first comes into contact.

In Fig. 2, the liquor level at the top of the crystallizing space or chamber 3*a*, is fixed by the height of an overflow outlet 49 which discharges the overflowing bath liquor passing through a depending pipe 50 into a seal pot 51. The latter is located in the tar chamber of a tar separator 24*a* generally like the tar separator 24 of Fig. 1. The pump 10 which passes liquor to the spray nozzles 7, 13 and 15 of Fig. 2, has its inlet connected to the chamber 24*a* at the clear liquor side of a vertical baffle 25 which divides the upper portion of the space into a tar chamber and a clear liquor chamber. The tar chamber has a valved tar outlet 27. The separate overflow re-

ceptacle 28 of Fig. 1 is omitted in Fig. 2, however. The overflow outlet 26a from the clear liquor chamber of the tar separator 24a may pass liquor into the mother liquor reservoir (not shown). In Fig. 2, crystals along with carrying liquor are withdrawn from the lower portion of the crystallizing space 3a to outlets 52, each connected to the suction pipe 53 of a corresponding slurry pump 54 which is arranged to pass the crystals and carrying liquor withdrawn, to crystal separator apparatus (not shown), as the pump 37 passes crystals and carrying liquor to the crystal separator 39 of Fig. 1.

The apparatus shown in Fig. 2 also differs from that shown in Fig. 1 in having a circulating pump 55 with an inlet pipe 56 extending through the wall of the chamber 3a and having its open upper end centrally disposed in the space 3a at a level a short distance below the overflow level through the outlet 49. The discharge pipe 57 of the pump 55 opens upwardly into the lower end of the hopper bottom portion of the chamber 3a. The pump 55 with its inlet and outlet connections forms an effective and regulable device for materially augmenting the recirculation effects of the pumps 10 and 30 of Fig. 2. With the pump 55 delivering a relatively strong, upwardly directed stream into the lower end of the chamber 3a, the smaller and lighter ammonium sulphate crystals suspended in the bath liquor, are largely kept away from the crystal outlets 52 in which the larger crystals are drawn, since the upflowing stream discharged through the pipe 57 exerts much greater opposition to the settling movement of small crystals than to the settling movement of the large crystals.

Except as already indicated, the operation of the apparatus shown in Fig. 2 is essentially like that of the apparatus shown in Fig. 1.

Fig. 3 illustrates apparatus advantageously different structurally and operatively from the apparatus shown in Figs. 1 and 2, in the following important respects:

The apparatus shown in Fig. 3 has its scrubbing space or chamber and its crystallizing space or chamber in separate tanks or receptacles; the Fig. 3 apparatus includes means for spraying finely divided liquor into one portion of the scrubbing chamber space and for spraying less finely divided liquor into another portion of said space which is so located that the coarser spray will absorb most of the fine spray entrained by the gas stream and thus minimize the amount of entrained liquor carried out of the scrubbing chamber by the outflowing gas; the apparatus shown in Fig. 3 includes a simplified and improved pumping arrangement; and includes liquor heating means by which the formation of small crystals and the growth of large crystals are subject to a desirable control.

The scrubbing chamber or space 2b of Fig. 3 comprises the entire space enclosed by a cylindrical, vertically disposed tank 60 with its lateral gas inlet 5 and gas outlet 6 respectively adjacent the lower and upper ends of the tank. The liquor sprayed into the chamber 2b does not accumulate therein, but passes through a bottom outlet opening into a liquor discharge pipe 61, and passes through the latter into the open upper end of a crystallizing tank or receptacle 62 kept substantially filled with bath liquor. The pipe 61 comprises a depending loop portion providing a liquid seal between the scrubbing chamber 2b in which the gas pressure is greater than the pressure of the atmosphere into which the discharge end of the pipe 61 opens. The pipe 61 is surrounded by a steam jacket 61'. The primary purpose of the pipe 61 is that of a conduit, but the desupersaturation of the ammonium sulphate liquor supersaturated in the scrubbing chamber 2b, is initiated in the pipe 61, and the latter may be regarded as an initial crystallizer or pre-crystallizer.

The main crystallizer tank or receptacle 62 is structurally similar to, and serves the tar separating purposes of the tar separator 24a of Fig. 2. The receptacle 62 includes a baffle 63 analogous to the baffle 25 of Fig. 2. The baffle 63 separates a clear liquor outlet compartment at its right from a tar receiving compartment at its left. The scrubber outlet pipe 61 discharges into said receiving compartment, as do the liquor return pipes 75 and 82 hereinafter described. Tar collecting in the upper end of the receiving compartment may be periodically removed by a manual skimmer, or by the use of a valved outlet (not shown), like the outlet 27 shown in Fig. 1 and in Fig. 2. Some of the liquor passing into the crystallizing receptacle 62 is withdrawn from the clear liquor compartment of the latter by a pump 10, and crystals settling in the lower, hopper bottom portion of the receptacle 62, are withdrawn along with carrying liquor by the slurry pump 68. Some of the liquor withdrawn from the receptacle 62 by the pump 68 is sprayed into the lower portion of the scrubbing chamber 2b, as is hereinafter described.

The pump 10 passes liquor withdrawn from the receptacle 62 through a pipe 9 including a liquor heater 64, to a spray device 7b, shown as comprising a single spray nozzle axially disposed in the chamber 2b, adjacent the upper end of the latter. The pump 10 also passes liquor from the tank 62 through the pipe 9, heater 64 and branch pipe 65 to a spray device 66 for spraying liquor into the scrubbing chamber 2b at a level substantially below the level of the spray device 7b.

In the form shown in Fig. 3, the spray device 66 comprises a bustle pipe surrounding the tank 60 and supplying liquor to a plurality of spray nozzles 67 extending through the tank wall and arranged to discharge sprays directed downwardly and toward the axis of tank 60. In the preferred form of the invention, the liquor sprayed into the chamber 2b through the nozzles 67 is much more finely subdivided than the liquor sprayed into the upper end of the chamber 2b through the nozzle 7b. In practice, the nozzles 67 are advantageously of one or another of the types used to "atomize" water and other liquids, and are adapted to subdivide the liquor which they spray into the chamber 2b into particles having an average diameter of less than one-half milimeter. As has been explained in my prior application Serial No. 3,133 filed on January 19, 1948, for the use of the general method disclosed and claimed herein, it is important that the spray nozzles employed are of the non-blocking type, so that they will not be blocked up by the crystal slurry pumped to them from the bottom of a container, such as the tank 1, or the crystallizer tanks 62, 62C or 3d of Figs. 3, 4 and 5, respectively. For example, the well known and widely used spray nozzles of the construction type shown in the Eneas Patent 1,101,264 of June 23, 1914, are well suited for this purpose. Preferably, the nozzle 7b, which may also be of well known form, is adapted to discharge liquor in the form of a relatively coarse spray consisting

of particles having an average diameter of about two millimeters. The inlet spray nozzle 73 is advantageously one adapted to produce a relatively coarse spray.

In practice, the difference between the level of the nozzles 67 and the level of the nozzle 7b may be of the order of ten or fifteen feet, so that the pressure at which the liquor is supplied to the nozzles 67 exceeds the pressure at which the liquor is supplied to the nozzle 7b by several pounds per square inch. This difference in the supply pressures facilitates the maintenance of the desired difference in the size of the liquor droplets discharged by the nozzles at the different levels.

The rapidity with which ammonia is removed from gas into which liquor containing ammonia sulphate and sulphuric acid is sprayed, rapidly increases as the size of the liquor spray particles is reduced. The fine spray discharged by the nozzles 67 is thus a more efficient agent for removing ammonia from the gas stream, than is the coarser spray discharged by the nozzle 7b. The amount of spray liquor which the gas stream tends to entrain also increases rapidly as the size of the spray liquor particles decreases. In consequence, if all of the liquor sprayed into the scrubbing chamber were as finely divided as is the liquor sprayed through the nozzles 67, the amount of entrained liquor which the gas would carry out of the scrubbing chamber through its outlet 6, would be excessive. With the arrangement shown in Fig. 3, however, the coarse liquor spray particles discharged by the nozzle 7b and moving along distributed paths through the portion of the gas stream between the level of the nozzles 67 and the outlet 6, absorb or coalesce with most of the fine spray particles entrained by the adjacent upflowing gas, and are thus knocked out of or removed from the ascending gas stream before they reach the outlet 6.

The steam of coarse spray particles discharged into the ascending gas stream at one level, and the stream of finer spray particles discharged into the gas stream at some lower level, thus cooperate to insure a desirably high ammonia absorbing capacity, coupled with a desirably low rate at which entrained liquor is moved out of the scrubbing chamber by the gas. The simultaneous discharge of a plurality of spray streams into the scrubber space at a corresponding plurality of portions of the scrubber space displaced from one another along the length of the path of gas flow through said space increases the efficiency with which the ammonia content of a given volume of gas can be removed by a relatively small amount of spray liquor, regardless of whether the different spray devices discharge fine spray or coarse spray. Thus, I have found experimentally that when a relatively moderate amount of spray liquor is sprayed into the scrubber chamber in three generally similar spray streams, at three different levels, about ninety-two percent of the ammonia in the gas is removed by the lowermost spray, about ninety-nine percent of the ammonia in the gas is removed by the conjoint action of the two lowermost sprays, and that the three sprays collectively remove 99.75 percent of the ammonia content of the gas.

An increase in the ammonia absorbing capacity of the liquor sprayed into the scrubbing chamber, contributes to a decrease in the amount of such liquor required to absorb the ammonia, and permits a reduction in the bulk, and in the construction and operating costs of the apparatus

required to recover the ammonia in a given volume of gas.

As previously noted, some of the liquor withdrawn from the crystallizer 62 by the pump 68 is sprayed into the scrubber chamber 2b. Thus, as shown in Fig. 3, liquor and crystals drawn from the crystallizer by the pump 68 are passed through the pump discharge pipe 69 to a classifier 70. The latter has an outlet connected through a valve 71 and pipe 72 to a spray nozzle 73 discharging into the lower end of the scrubbing chamber 2b. As diagrammatically shown, the nozzle 73 is located in the gas inlet passage 5 in position to discharge a spray into the lower portion of the chamber 2b.

The classifier 70 is a solid and liquid separating device which may be of the cyclone type and form largely used in the metallurgical art to separate finely divided solid matter from water. The classifier 70 of Fig. 3 normally operates to pass the major portion of the entering crystals into the classifier from the pump 68 through the classifier outlet 74 to a subjacent crystal separator 39. The latter may be of a conventional type customarily employed to separate ammonium sulphate crystals from carrying liquor. The relative amounts of liquor passing from the classifier 70 to the nozzle 73 and to the separator 39 may be regulated by relative adjustments of the valves 71 and 74. The adjustment of the valve 71 also regulates the pressure at which liquor is passed into the spray nozzle 73. As shown, the pipe 72 comprises a depending loop portion 72' forming a liquid seal between the classifier 70 and the scrubbing chamber 2b.

As diagrammatically shown, the liquor separated from the crystals in the separator 39 passes from the upper portion of the separator back into the crystallizer 62 through a return pipe 75. As shown, the pipe 75 includes a liquor heater 76, the purpose of which is hereinafter stated. The pipe 75 also serves to return liquor to the crystallizing chamber from the crystal dryer 77. The latter may be a continuous dryer of conventional type arranged to receive crystals and accompanying liquor from the bottom of the separator 39 through a normally open valve 78. When the latter is closed, the crystals and carrying liquor may also be returned to the crystallizer chamber 62 from the lower portion of the separator 39 through a normally closed valve 79 and pipe 80. Ordinarily, the valves 78 and 79 are respectively closed and open to pass crystals and liquor back to the crystallizer 62 only when the average size of the crystals settling into the lower portion of the separator 39 is smaller than is required to satisfy the existing crystal demand. The crystals then returned through the pipe 80 are given time and opportunity for further growth in the crystallizer chamber 62.

In Fig. 3, a conventional acid catcher of the cyclone separator type has its inlet connected directly to the outlet 6 of the scrubbing chamber 2b. The acidified liquor separated from the gas in said acid catcher 81 passes through the valved bottom outlet 83 and pipe 82 to the crystallizer chamber 62. Ordinarily and as shown, the pipe 82 includes a depending loop portion providing a liquid seal which prevents gas from blowing out of the separator 81 through the pipe 82. The gas leaves the acid catcher through the outlet pipe 84 which has its inlet end within the acid catcher and adjacent the lower end of the latter.

As those skilled in the art understand, the formation of minute so-called seed crystals may

result from various changes in operating conditions, such for example as an increase in the rate at which ammonia is absorbed by the bath liquor in the scrubbing space, or a decrease in the temperature of the liquor. The over-production of seed crystals contributes directly to a reduction in the rate of crystal growth in the crystallizing chamber, and hence in the average size of the crystals produced.

The heaters 64 and 76 may be of any usual or suitable type. For example, they may be electric resistance heaters, but as diagrammatically shown, they are heaters of the tubular heat exchanger type with provisions for passing steam at regulable rates into their heating fluid spaces. The heater 64 forms a means for dissolving more or less of the small crystals entrained by the liquor drawn out of the crystallizing chamber 62 and moved through the heater 64 by the pump 10, and thus reduces the number of small crystals passed into the scrubbing chamber through the spray nozzles receiving spray liquor through the heater 64. The heater 76 may be used to dissolve more or less of the relatively small crystals returned from the separator 39 to the crystallizer 62 through the pipe 75. The dissolving action of small crystal content by the heating units 64 and 76 contribute to a reduction in the number of small crystals in the crystallizer 62, and thereby augments the crystal growth occurring in that chamber. In lieu of, or in addition to using the heaters 64 and 76, live steam may be injected into the corresponding liquor streams. Thus, as shown in Fig. 3, steam may be injected into the pipe 75, when and as desired, through a valved steam pipe 76'.

As diagrammatically shown in Fig. 3, make-up acid is fed through a pipe 11B into the portion of the crystallizer 62 immediately adjacent the outlet from the crystallizer to the pump inlet 10. Hot water alone, or in admixture with bath liquor for rock salt elimintaion or other cleaning purposes, may be passed into the inlet of the pump 10 through a valved pipe connection 16B and into the classifier 70 through a supply pipe 70'.

The apparatus shown in Fig. 3 is characterized by its mechanical simplicity. In addition to other advantages of the apparatus shown in Fig. 3, previously mentioned, the cost of construction of a scrubbing chamber 60 of large capacity, is reduced by making the enclosing tank in the form of a simple cylinder, rather than by giving it a tapered form such as is given the tanks 1 and 1a of Figs. 1 and 2, respectively. The cylindrical form of the tank 60 permits of an advantageous disposition in the tank of a suitable multiplicity of spray devices, and is less expensive to construct in large size units than is the tapered tanks shown in Figs. 1 and 2.

More or less of the crystal entraining liquor withdrawn from the scrubbing chamber 2b by the pump 68, may be returned to said chamber through the nozzle 73 without making use of the classifier 70. Thus as is shown in Figure 3A, the upper ends of the pipes 72 and 69 may be directly connected through the valve 71, and the pipe 69 may also be connected to the valve 74 for the passage of crystals and liquor into the separator 39. The supply to the lower Fig. 3 spray nozzle 73, of liquor having an acid content substantially the same as the acid content of the liquor passing away from the scrubbing space, and hence appreciably lower than the acid content of the liquor supplied to the more elevated spray nozzles of Fig. 3 in consequence of the inclusion in the last mentioned liquor of the make up acid supplied through the pipe 11B, is advantageous for two reasons. First, owing to the relatively high ammonia content of the gas when it first enters the scrubbing space of Fig. 3 through the gas inlet 5, the absorption of the ammonia by the liquor sprayed into contact with the gas would result in an undesirably rapid supersaturation of the contacting spray liquor and the formation of an undesirably large number of small new crystals, if the acid content of the contacting liquor were appreciably higher than the acid content of the liquor passing out of the scrubbing space. Second, the higher acid content in the liquor sprayed through the nozzles 7B and 67 into contact with gas from which a substantial portion of its initial ammonia content has been absorbed, reduces the amount of liquor discharged through the last mentioned nozzles which is needed to suitably lower the ammonia content of the gas leaving the scrubbing space. The discharge of a relatively coarse spray through the nozzle 73 makes the supersaturation effect less pronounced than it would be if the spray were finer.

The form of the invention shown in Fig. 4 closely resembles that shown in Fig. 3, except that in Fig. 4 only a single liquor pump is used. In Fig. 4 the pump 68 serves the same purposes served by it in Fig. 3, and serves in addition to supply liquor to the inlets of the spray devices 7b and 67. Liquor and entrained crystals or slurry, withdrawn from the crystallizer 62C of Fig. 4 by the pump 68, pass through the discharge pipe 69 to the classifier 70 and pass from the latter through a lower valved outlet 74 and through an upper outlet valve 71 and pipe 72, as in Fig. 3. In Fig. 4, however, the pipe 72 in addition to passing liquor to the spray nozzle 73, passes liquor through a branch pipe 85 and associated distributing means to spray nozzles 7b and 67. The arrangement of the latter, shown in Fig. 4, is like that shown in Fig. 3. Advantageously, however, the portion of the pipe 72 individual to the nozzle 73, includes a throttling valve 86.

The branch pipe 85 of Fig. 4 discharges into a classifier 87, which may be a counterpart of the classifier 70. The bottom outlet from the classifier 87 is connected through a valve 88 to branch pipes 89 and 90. The pipe 89 supplies spray liquor to the nozzle 7b, and the pipe 90 supplies spray liquor to the bustle pipe 66 and thereby to the spray nozzles 67. The branch pipes 89 and 90 include regulating valves 91 and 92, respectively. The valve 88 is shunted by a by-pass connection which includes valve 93 and a liquor heater 94, and a pipe 95 connecting the heater 94 to the connected ends of of the valves 88, 91 and 92. By suitable adjustment of the valves 71, 86, 88, 91, 92 and 93, the relative amounts of liquor passed to the nozzles 7b and 73 and to the group of nozzles 67, may be varied as desired. Furthermore, the pressures at the inlets to the nozzles 7b and 73, and at the inlet to the bustle pipe 66, may be varied as conditions make desirable. The heater 94 forms a means for controlling the amount of small crystals passing into the scrubbing chamber 2b through the nozzles 7b and 67. Instead of using the heater 94, live steam may be passed into the pipe 95 through the valved pipe connection 95'.

The pump simplification of Fig. 4 is thus attended by a desirable flexibility of control of the relative amounts of spray liquor supplied to the various spray nozzles, and the pressures at which

the liquor is supplied to the nozzles. The apparatus shown in Fig. 4 also differs from that shown in Fig. 3 in the omission of the crystallizer baffle 68 which may also be omitted from the Fig. 3 arrangement. As shown in Fig. 4, hot water, or hot water admixed with liquor may be passed to the inlet of the pump 68 through a pipe 16C, and make-up sulphuric acid may be supplied through a pipe 11C which opens into the hopper bottom portion of the crystallizer 62C, adjacent the outlet from the crystallizer connected to the inlet of the pump 68. The general operation of the apparatus shown in Fig. 4 may be like the apparatus shown in Fig. 3, except as already noted.

In Fig. 5 I have illustrated a relatively simple and compact structural arrangement of apparatus for converting the ammonia in distillation gas into ammonium sulphate, in which the scrubbing chamber 2*d* and the crystallizing chamber 3*d* are in separate receptacles as in Figs. 3 and 4. The scrubbing chamber 2*d* of Fig. 5 is enclosed by a cylindrical tank 100, having a hopper bottom and a depending coaxial liquor outlet pipe 101. The latter is open at its upper end to the chamber 2*d* and has its lower end submerged in the liquor held in the receptacle 102. The latter is open at its upper end and forms the surrounding and bottom walls of the crystallizing chamber 3*d*. The pipe 101 and chamber 102 cooperate to form a liquor sealed connection between the lower ends of the scrubbing chamber 2*d* and crystallizing chamber 3*d*. The wall of the chamber 3*d* is made hollow to serve as a steam heating jacket space 103 for said chamber. Similarly, the output pipe 101 is formed with a double wall to provide an annular steam heating space 104.

Liquor is withdrawn from the crystallizing chamber 3*d* at an upper level, by a pump 10 which passes the liquor to the scrubbing chamber 2*d* through a spray nozzle 7*d* carried by the depending outlet end portion of the pump discharge pipe 105. A depending upper end portion of the pipe 105 passes centrally into the upper end of the chamber 2*d* through the top wall 106 of the tank 100. Make-up acid may be passed into the inlet of the pump 10 through a valved pipe 11*d*. As shown, the inlet of the pump 10 is connected to a ring shaped trough 107 extending horizontally around the vertical axis of the chamber 3*d*. Liquor overflows from the crystallization receptacle 102 through an overflow pipe 108 which discharges into a tar separator 109 which as shown, is similar in type to the tar separator 24*a* of Fig. 2, and is provided with a similar valved tar outlet 27. In Fig. 5, the bottom outlet of the tar separator is connected to the inlet of the pump 110 which returns liquor to the upper end of the chamber 3*d* through a pipe 111.

A pump 112 draws liquor from the clear liquor chamber of the tar separator 109, and passes it through a valve 113 and pipe 114 to a spray ring 115 carried at the underside of the upper top plate 106 of the tank 100. The spray ring 115 discharges liquor against the inner wall of the upper portions of the scrubbing chamber 2*d*, and against the outer surface of the portion of the pipe 104 extending downward from the underside of the top wall plate 106. As shown, a branch from the pipe 114 supplies liquor to a nozzle 116 which discharges liquor against the inner wall surface of the tank 100 adjacent its gas outlet 6. Another pump 117 draws liquor from the clear liquor chamber of the tar separator and discharges it into the lower portion of the scrubbing chamber 2*d* through a spray valve 73' which may be similar in location, form and general operative effect to the spray valves 73 of Figs. 3 and 4. Hot water may be passed into the pipe 114 through a valved pipe 16*d* opening to the pipe 114 at a level above the valve 113. Hot water may also be passed into the nozzle 73' through a valved pipe 16*e*. The operation of the apparatus shown in Fig. 5 may be generally like that of the apparatus shown in Figs. 3 and 4 except as a result of the omission from Fig. 5 of fine spray nozzles, like the nozzles 67 of Figs. 3 and 4, below the central upper nozzle 7*d*.

The exothermic reaction in which free ammonia combines with acidified ammonium sulphate liquor used in recovering ammonia from coke oven or analogous distillation gas, results in the evaporation in the scrubbing space of any of the forms of apparatus shown, of some of the water present in such liquor. Such evaporation tends to increase the supersaturation of the unevaporated residue of the liquor. The pressure and temperature in the scrubbing space are higher than the pressure and temperature respectively, of the ambient atmosphere. In consequence, there is an evaporation into the ambient atmosphere of some of the water content of the ammonium sulphate liquor. The effect of such evaporation to the atmosphere of liquor passing into the crystallizer shell and at the upper surface of the liquor therein as shown in Figs. 3, 4 and 5, contributes to a desirable reduction in the temperature and in the ammonium sulphate content of liquor desupersaturated in the open crystallizing chamber.

In all of the forms of the invention illustrated, the liquor in the portion of the crystallizer space immediately adjacent the outlet from which the liqpor to be sprayed to the scrubbing space is withdrawn, is ordinarily in its normally saturated condition. However, as a result of variations in operating conditions, the liquor withdrawn for spraying purposes may be occasionally slightly undersaturated or slightly supersaturated. In regular operation, however, the undersaturation is negligibly small and the extent of supersaturation is much less than that of the liquor returned to the crystallizer from the scrubbing space. The average condition of the liquor withdrawn for spraying purposes may thus be designated as an approximately saturated condition, and is so designated in some of the appended claims.

In each of my prior applications, Serial No. 668,392, filed May 9, 1946; Serial No. 659,444, filed April 4, 1946; Ser. No. 674,334, filed June 4, 1946; Ser. No. 720,593, filed January 7, 1947; Ser. No. 771,196, filed August 29, 1947, all now abandoned; and Ser. No. 3,133, filed January 19, 1948, I have disclosed subject matter which is claimed generically or specifically in the present application, which is a continuation in part of each of said prior applications.

While in accordance with the provisions of the statutes, I have illustrated and described the best forms of embodiment of my invention now known to me, it will be apparent to those skilled in the art that changes may be made in the forms of the apparatus disclosed without departing from the spirit of my invention as set forth in the appended claims, and that in some cases certain features of my invention may be

used to advantage without a corresponding use of other features.

Having now described my invention, what I claim as new and desire to secure by Letters Patent is:

1. The method of continuously producing sulphate of ammonia which comprises passing into a substantially unobstructed scrubbing space a gas containing free ammonia, contacting such gas with saturated ammonium sulphate solution containing free sulphuric acid, withdrawing ammonium sulphate liquor to make up such solution from a body of ammonium sulphate liquor in a crystallizing space below said scrubbing space, said contacting being effected by spraying by whirling motion and under such pressure so as to disperse said solution in fine spray over substantially the whole cross sectional area of the space and permit solid crystalline particles and supersaturated liquid spray to fall into said body whereby the growth of ammonium sulphate crystals in said body is promoted, withdrawing crystals from said crystallizing space and adding sulphuric acid to the liquor to make up for the acid combining with ammonia in the scrubbing space.

2. A method as specified in claim 1, in which the liquor introduced into said scrubbing space entrains ammonium sulphate crystals.

3. A method as specified in claim 1, in which the liquor dispersed throughout said scrubbing space is in the form of spray particles having an average diameter of less than two millimeters.

4. A method as specified in claim 1, in which the acid content of the liquor sprayed into the scrubbing space adjacent such gas inlet is about 5 or 6 per cent by weight, and in which the acid content of the liquor sprayed into said scrubbing space more remote from said inlet exceeds the first mentioned acid content.

5. In the continuous production of sulphate of ammonia, the method which consists in moving gas having a small ammonia content through a scrubber space between a lower gas inlet to, and an upper gas outlet from said space, passing acidified ammonium sulphate liquor in the form of a coarse spray into a portion of said space and passing acidified ammonium sulphate liquor in the form of a relatively fine spray into a portion of said space nearer to said inlet than the first mentioned portion, said coarse spray consisting of liquor particles having an average diameter not less than two millimeters and said fine spray consisting of fine particles having an average diameter of not greater than one-half millimeter.

6. In the continuous production of sulphate of ammonia, the method which consists in moving gas having a small ammonia content through a scrubber space between a lower gas inlet to, and an upper gas outlet from said space, passing acidified ammonium sulphate liquor in the form of a relatively coarse spray into an upper portion of said space and passing acidified ammonium sulphate liquor in the form of a relatively fine spray into an intermediate portion of said space, and spraying liquor in the form of a relatively coarse spraying into a lower portion of said space adjacent said gas inlet.

7. The method of continuously producing sulphate of ammonia which comprises passing into a substantially unobstructed scrubbing space a gas containing free ammonia, contacting such gas with saturated ammonium sulphate solution containing free sulphuric acid and entraining solid crystalline particles and reacting with the free ammonia to form supersaturated ammonium sulphate liquor, withdrawing supersaturated ammonium sulphate liquor from said scrubbing space and desupersaturating the latter to make up all of said solution used in carrying out the method from a body of ammonium sulphate liquor in a crystallizing space below said scrubbing space, said contacting being effected by spraying under such pressure so as to disperse said solution in fine spray over substantially the whole cross sectional area of the space and permit solid crystalline particles and supersaturated liquid spray to fall into said body whereby the growth of ammonium sulphate crystals in said body is promoted, withdrawing crystals from said crystallizing space and adding sulphuric acid to the liquor at a rate so related to the rate at which the ammonia containing gas is passed into said scrubbing space as to effect the reaction of substantially all of the ammonia in the gas with the acid, and make up for the acid combining with ammonia in the scrubbing space.

8. A method as specified in claim 7, in which the gas passes first through an inlet portion and thence through an outlet portion of said scrubbing space, and including the step of adding make-up acid mainly to the liquor sprayed into the last mentioned portion of the scrubbing space.

9. In removing ammonia from coke oven gas by spraying acidulated sulphate of ammonia liquor into contact with the gas in a scrubbing space surrounded by a wall formed of acid resisting plate, the method which consists in spraying liquor into said space to disperse the sprayed liquor therein, and to maintain a layer of supersaturated liquor on the inner side of said wall, and applying heat to the outer side of said wall to lower the saturation point of the liquor in said layer and thereby prevent salt incrustations on the inner side of the wall.

10. By-product coke oven apparatus for the production of ammonium sulphate crystals, comprising structure enclosing a scrubbing chamber having a gas inlet adjacent its lower end and a gas outlet adjacent its upper end, and a liquor outlet below the level of said gas inlet, spraying devices arranged in said chamber at different levels, a crystallizer comprising a liquor container and a pipe bent to form a liquor seal and having one end portion connected to the lower end of said chamber to receive liquor therefrom and having its second end portion above said container and arranged to discharge liquor into the latter and having an intermediate portion in the form of a depending loop, a pump having an inlet connected to said liquor container for withdrawing liquor and crystals entrained therein from said container and having a discharge pipe line connected to and discharging liquor and crystals through said spraying devices, a separator device for separating crystals from liquor, a branch pipe including a regulating device connecting said pipe line to said separator device, and means for admixing sulphuric acid with the liquor to be supplied to the spraying devices.

11. Apparatus as specified in claim 10 including a crystal classifier having an inlet connected to said discharge pipe line and having an outlet connected to and passing liquor and crystals to at least one of said spray devices, and having a second outlet for the discharge of crystals not passed to any of said spraying devices.

12. By-product coke oven apparatus for the production of ammonium sulphate crystals, comprising structure enclosing a scrubbing chamber having a gas inlet adjacent its lower end and a

gas outlet adjacent its upper end and a liquor outlet below the level of gas inlet, spray devices arranged in said chamber at different levels, a crystallizer comprising a liquor container and a pipe bent to form a liquor seal and having one end portion connected to the lower end of said chamber to receive liquor therefrom and having its second end portion above said container and arranged to discharge liquor into the latter and having an intermediate portion in the form of a depending loop, a first pump having an inlet connected to said liquor container for withdrawing liquor therefrom and having a discharge pipe line connected to and discharging through one of said spray devices, a second pump having an inlet connected to said liquid container for withdrawing liquor therefrom at a level different from that at which the inlet of the first pump is connected to said container, and having a discharge pipe line connected to and discharging through another of said spraying devices at a level different from the level of the spraying device through which the first pump discharges, a separator device for separating crystals from liquor, and a pipe connecting the discharge pipe line of one of said pumps to said separator for passing liquor and crystals into the latter.

13. In a coke oven by-product plant including apparatus for producing ammonium sulphate crystals by scrubbing coke oven gas containing ammonia with an acidified ammonium sulphate solution, the combination with structure formed with a crystallizing space and a substantially unobstructed scrubbing chamber, the latter having a lower liquor and crystal outlet arranged to discharge into said space, and having a gas inlet and a gas outlet each spaced away from the other and from said liquor and crystal outlet, nozzle means comprising first and second non-blocking spray nozzles mounted in said structure and arranged to discharge a liquor spray and crystals into portions of said chamber respectively adjacent and remote from said gas inlet, conduit means connected to said structure and communicating with said chamber for passing coke oven gas through said gas inlet and for withdrawing gas from said chamber through said gas outlet, means connected to said structure for withdrawing saturated ammonium sulphate liquor and crystals from said crystallizing space and spraying said liquor and crystals into said chamber comprising one pump having an inlet conduit connected to said crystallizing space at one level and an outlet conduit connected to said second nozzle, and comprising a second pump having an inlet connected to said crystallizing space for withdrawing liquor therefrom at a level lower than the first mentioned level and having a discharge pipe line connected to and discharging through said first nozzle, a separator device for separating crystals from liquor, and a pipe connecting said discharge pipe line to said separator for passing crystals and liquor into the latter, and means for adding make-up sulphuric acid to the liquor passing from said crystallizing space to said nozzle means.

14. In a coke oven by-product plant including apparatus for producing ammonium sulphate crystals by scrubbing coke oven gas containing ammonia with an acidified ammonium sulphate solution, the combination with structure formed with a crystallizing space and a substantially unobstructed scrubbing space, and including wall structure surrounding said crystallizing space and said scrubbing space, non-blocking spray nozzles mounted in said structure, said scrubbing space having a lower liquor and crystal outlet arranged to discharge into said crystallizing space, and having a gas inlet and a gas outlet each spaced away from said nozzles, each of said nozzles being arranged to discharge a liquor spray and crystals into an adjacent portion of said scrubbing space, said nozzles being arranged to disperse said spray over substantially the whole cross sectional area of said scrubbing space, gas conduit means connected to said structure and communicating with said scrubbing space for passing coke oven gas into said scrubbing space through said gas inlet and for withdrawing gas from said scrubbing space through said gas outlet, means connected to said structure for withdrawing saturated ammonium sulphate liquor and crystals from said crystallizing space and spraying said liquor and crystals into said scrubbing space and comprising pumping means having inlet conduit means connected to said crystallizing space for the withdrawal of liquor and crystals therefrom, and having outlet conduit means for discharging liquor and crystals withdrawn from the crystallizing space and including a separate conduit connection to each such nozzle, and means for adding make-up sulphuric acid to the liquor passing from said crystallizing space to said nozzle means, at a rate so related to the rate at which the gas containing ammonia is passed into said scrubbing space that substantially all of the ammonia in the gas reacts with the sulphuric acid to form ammonium sulphate liquor and crystals, whereby substantially all of the ammonium sulphate liquor sprayed into said scrubbing space is liquor which has been previously withdrawn from said scrubbing space.

15. Apparatus as specified in claim 14, in which said gas conduit means includes a gas supply pipe having a horizontal discharge end portion for passing a stream of coke oven gas horizontally through said gas inlet and through a portion of said scrubbing space adjacent to said gas inlet and in which a portion of the liquor spray discharged by said spray nozzles is passed into said scrubbing space portion adjacent said gas inlet.

16. Apparatus as specified in claim 14, including a pipe connected to said inlet conduit, means for connecting the latter to a source of cleaning fluid, and a valve in said pipe, whereby when said valve is open, cleaning fluid is sprayed into said scrubbing space through said non-blocking nozzles.

17. Apparatus as specified in claim 14, in which said structure includes a vertical tubular shell formed of acid resisting, metal plate material and constituting a wall surrounding said scrubbing space, and heating means external to said wall for passing heat into the outer side of said wall, whereby liquor spray discharged by said spray nozzles and supersaturated by contact with the ammonia containing coke oven gas and impinging against and forming a liquid film on the inner side of said wall, has its saturation point lowered, and salt incrustation on the inner side of the wall thereby prevented, by the heat passed into the wall by said heating means.

18. Apparatus as specified in claim 14, in which the structure forming the crystallizing space includes a vertically disposed, open ended pipe having its upper end open to receive ammonium sulphate liquor and crystals passing downward through said scrubbing space, and a receptacle of a larger horizontal cross section than said pipe and into which said pipe depends and which cooperates with said pipe to form a crystallizer

space external to said pipe and receiving liquor adjacent its lower end through the open lower end of said pipe, first and second conduit connections to said receptacle at respectively low and high levels, said first conduit connection forming a portion of said pumping means through which the latter passes liquor and crystals to said separator, and said second conduit connection forming a portion of said pumping means through which the latter passes liquor and entrained crystals to said spray nozzles, a second receptacle, and an overflow connection through which liquor in the first mentioned receptacle above said relatively high level overflows into said second receptacle.

19. In a crystallizing and reaction tank system for producing crystals of $(NH_4)_2SO_4$ by reaction of ammonia containing gas with H_2SO_4 solution, tank apparatus comprising a reaction tank section and a crystallizing tank section, said reaction tank section having an inlet for a conduit for passing the ammonia containing gas into an inlet portion of said reaction tank section, a spray nozzle with fluid rotating passage walls located adjacent said inlet so as to immediately contact the entering gas with spray reaction with the ammonia containing gas, a plurality of spray nozzles with fluid rotating passage walls located in an unobstructed portion of said reaction section, displaced from said inlet section, and arranged to cover the cross sectional area of said unobstructed portion with spray, and an outlet conduit for gas in said unobstructed portion, said crystallizing tank section being arranged to receive solution and crystals moving down from the bottom of said reaction tank section to said crystallizing tank section, a suspension conveying system including said crystallizing tank section, pumping means for conveying a suspension of crystals from a portion of said crystallizing tank section containing relatively large crystals to a crystal separating device, and conduit means connected to a portion of said conveying system which follows said portion containing relatively large crystals and which includes conduits through which suspensions of crystals are passed to the spray nozzles in the unobstructed portion of said reaction section and inlet means for the addition of fresh acid to said suspension conveying system.

CARL OTTO.

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The following references are of record in the file of this patent:

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Certificate of Correction

Patent No. 2,599,067 June 3, 1952

Carl Otto

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows:

Column 1, line 4, strike out "and sulphuric acid to thereby" and insert instead *by bringing coke oven gas or*; lines 6 and 7, for "ammonia" read *ammonium*; column 5, line 23, for "1 to 23" read *7 to 23*;

and that the said Letters Patent should be read as corrected above.

Signed and sealed this 17th day of August, A. D. 1954.

[SEAL]

ARTHUR W. CROCKER,
Assistant Commissioner of Patents.

(No Model.) 2 Sheets—Sheet 1

M. M. MONSANTO.

APPARATUS FOR THE VAPORIZATION OF LIQUIDS.

No. 441,106. Patented Nov. 18, 1890.

Fig. 2.

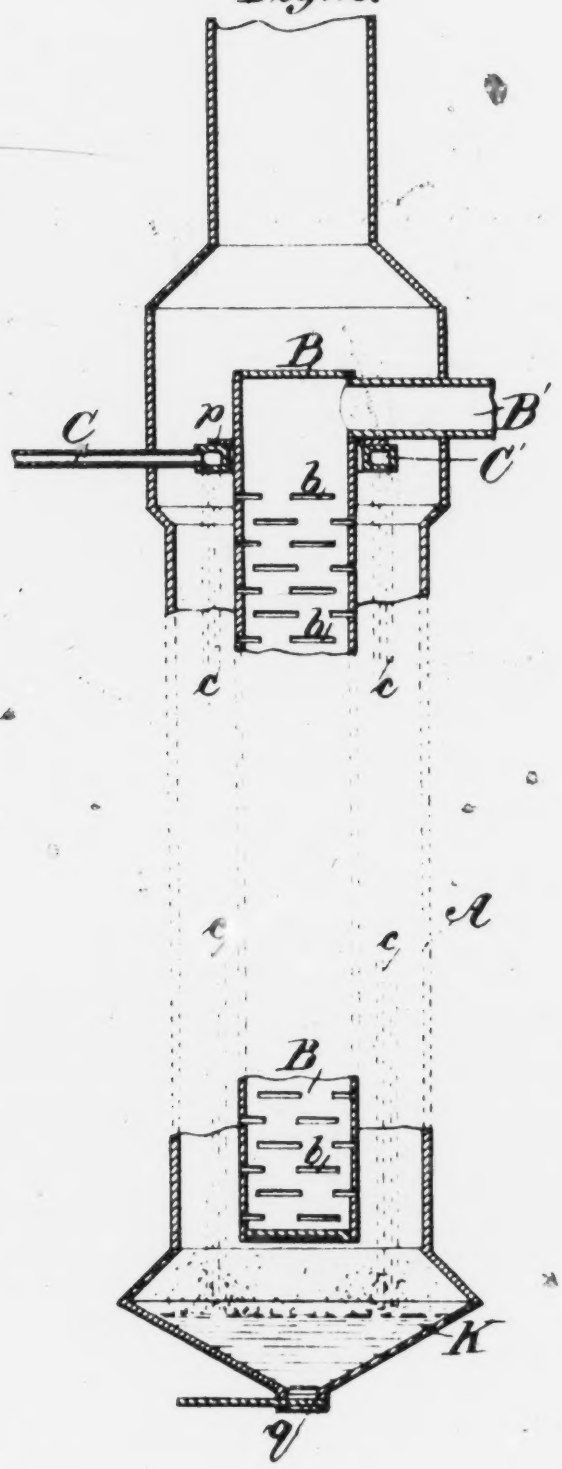


Fig. 1.

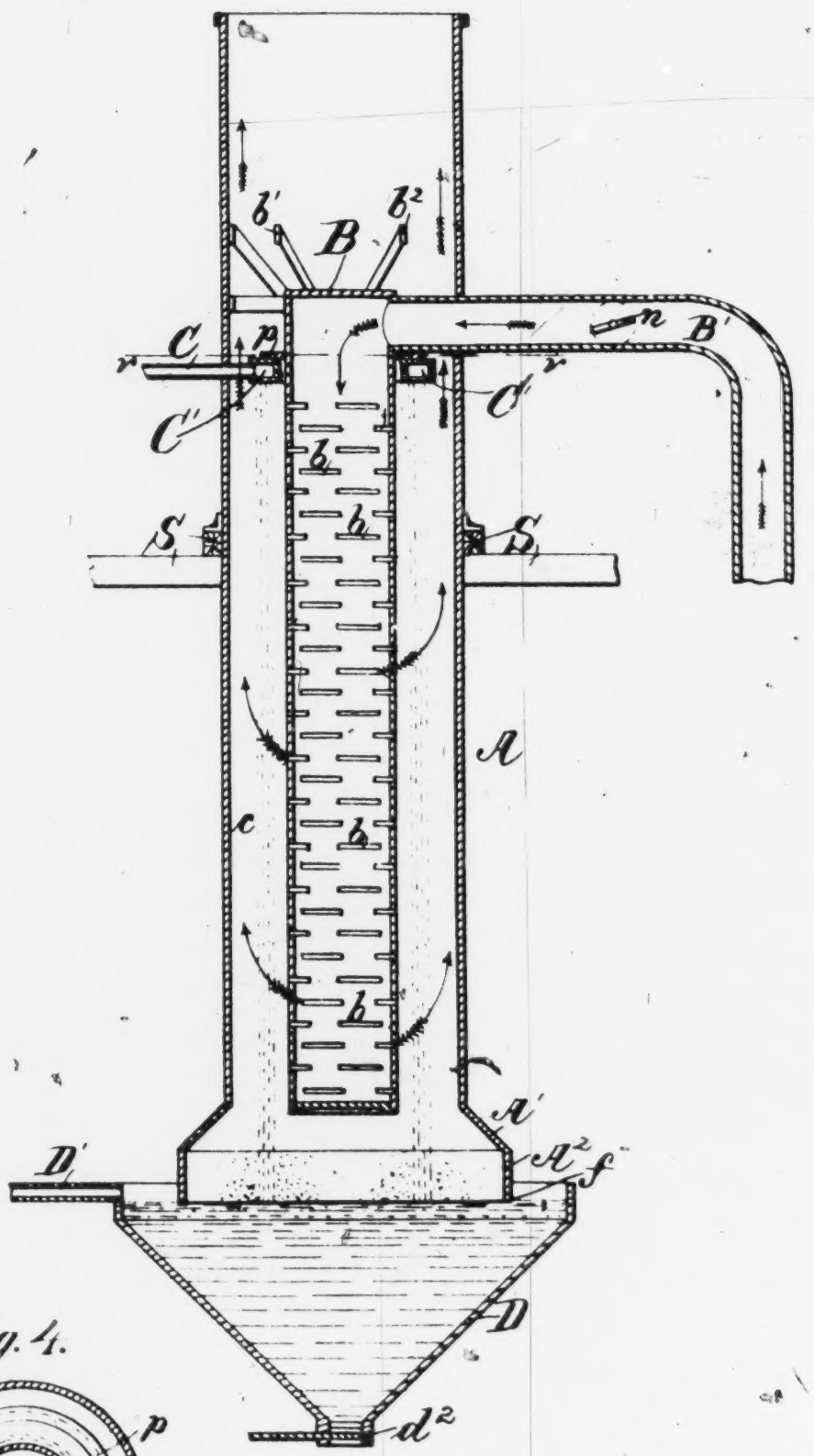
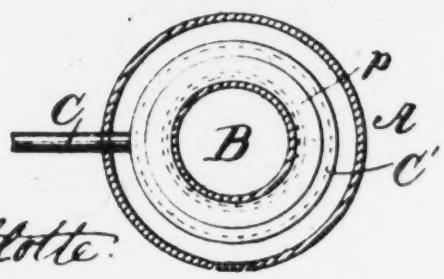


Fig. 4.



WITNESSES:

Edward F. Bartlett

Edward A. Baldwin

INVENTOR

Maurice M. Monsanto

BY

Lacey L. Stover

ATTORNEY

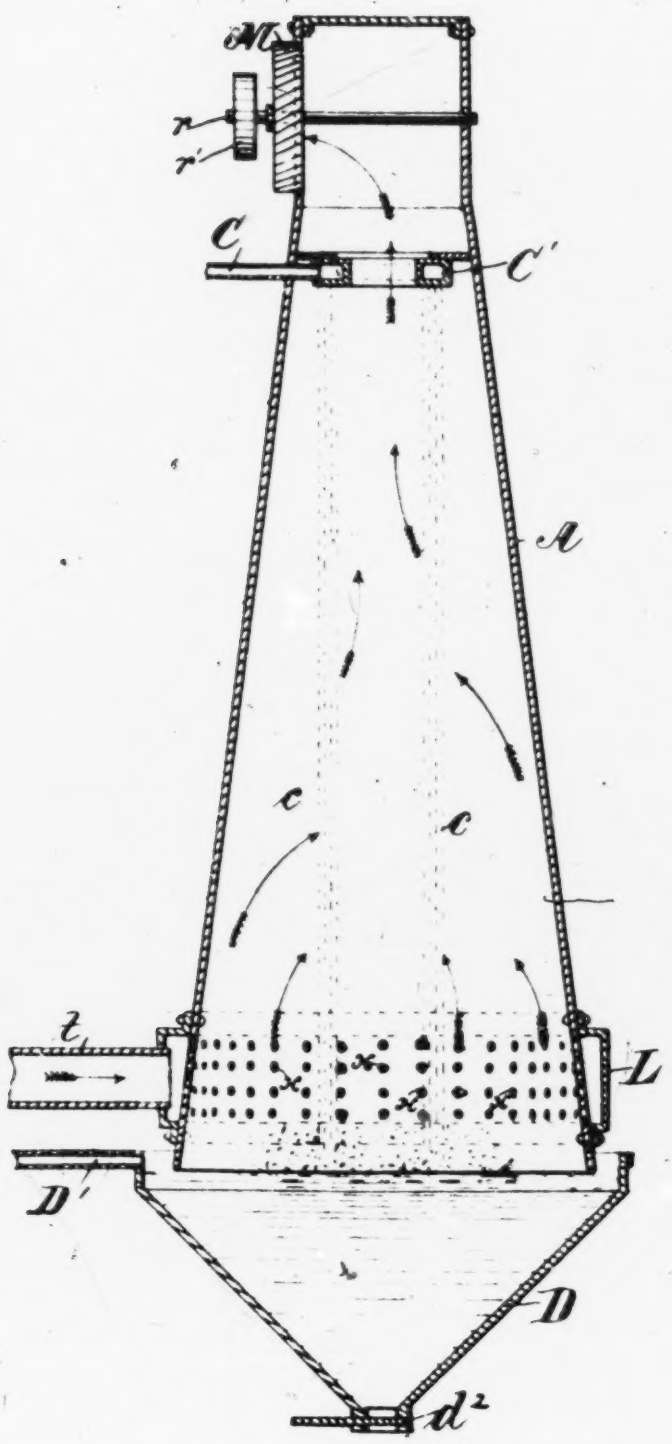
(No Model.) 2 Sheets—Sheet 2.

M. M. MONSANTO.

APPARATUS FOR THE VAPORIZATION OF LIQUIDS.

No. 441,106. Patented Nov. 18, 1890.

Fig. 3.



WITNESSES:
Edward H. Bartlett
Edward A. Baldwin

INVENTOR
Mariano M. Monsanto,
BY
Jacob L. Stover,
ATTORNEY

UNITED STATES PATENT OFFICE.

MAURICIO M. MONSANTO, OF HOBOKEN, NEW JERSEY, ASSIGNOR TO THE NATIONAL SALT AND CHEMICAL COMPANY.

APPARATUS FOR THE VAPORIZATION OF LIQUIDS.

SPECIFICATION forming part of Letters Patent No. 441,106, dated November 18, 1890.

Application filed October 25, 1888. Serial No. 289,150. (No model.)

To all whom it may concern:

Be it known that I, MAURICIO M. MONSANTO, of Hoboken, county of Hudson, and State of New Jersey, have invented certain new and useful Improvements in Apparatus for the Vaporization of Liquids, of which the following is a full, clear, and exact description, reference being had to the accompanying drawings, and to the letters of reference marked thereon.

My invention relates to apparatus designed for the vaporization of alkaline or saccharine liquids, in which a current of air or gases, preferably heated, is forced in intimate contact with a descending current of the liquid in a finely-subdivided condition, also preferably heated, whereby the air is made to absorb moisture from the liquid, saturating itself therewith, thereby concentrating or reducing the volume of the liquid to or near to the point of the crystallization of the contained solids. The air or gases for the purpose may be atmospheric or the purified products of combustion, as described in an application for another United States Patent filed October 25, 1888, Serial No. 289,151. The latter gases may be advantageously applied in the apparatus for the vaporization of saccharine and such other liquids which are deleteriously affected by the oxygen of the air, while the carbonic acid contained in the products of combustion will act favorably in carbonating any excess of free lime the liquids may contain or which in the course of manufacture may have been introduced for clarification. The sulphurous oxide may also act advantageously in bleaching the saccharine matter, it being now applied for that purpose.

The apparatus is very inexpensive, effective, and economical in its operation.

Reference is to be had to the accompanying drawings, forming part of this specification, in which similar letters of reference indicate corresponding parts in all the figures.

Figure 1 represents a vertical section of my improved apparatus. Fig. 2 represents a modification thereof. Fig. 3 represents still another form of the same. Fig. 4 is a cross section on line V V, Fig. 1.

In the drawings, A represents a vertical outer shell or chamber, herein indicated as cylindrical, (but it may be rectangular or any other suitable form,) surrounding, in Figs. 1 and 2, a central perforated shell or chamber B, which is closed at both ends. Into this central chamber B the evaporating air or gases are designed to be forced by means of a fan or other suitable device (not shown) through the conduit B′, which is provided with a regulating damper or valve *n*, and escape through the perforations or slots *b* and impinge on the liquid, which falls in the form of rain *c c* from the perforated distributing pipe or chamber C′, which is annular and fixed between the cylinders A B near the top of the latter. This distributing-pipe C′ is fed by the conduit C, and the space between the pipe C′ and the chamber B is closed by a baffle plate or flange *p*, so that the air or gases shall not move upward through this space, but be forced to pass up through and in contact with the falling drops of liquid.

The central shell or chamber B is secured in place centrally within the outer shell A, preferably by stays $b' b^2$, which are fixed above the annular pipe C′, so that the shower of the liquid may find no impediment in its descent upon which to make deposit.

The shells A and B are placed axially above a receiving-vessel D, of greater diameter than the bottom of the shell A, and open at the top and having a bottom discharge-gate d^3 and an overflow-pipe D′, the latter being at the level of the liquid in vessel D.

The shell A is expanded toward the bottom, as shown at A′ A^2, Fig. 1, and preferably just touches the liquid in vessel D at *f*, or it may be arranged a little above or below the surface of the liquid. This expansion or enlargement of cylinder A at the bottom is for the object of preventing the contact of the liquid upon the inside of the shell A when said liquid falls into the liquid contained in the vessel D. The crystals resulting from vaporization of the liquid will fall to the bottom of the vessel D, whence they may be removed through the gate d^2, when desired, and the overflowing liquid passing off through the pipe D′. The outlet from D is designed to be repumped into and through conduit C, with the addition of fresh liquid to compen-

sate for that previously evaporated, or the liquid overflowing from the vessel D may be conducted to a heating-vessel, (not shown,) from which it will be fed again through conduit C.

The vessel D may have heat directly applied to it to further heat and vaporize and concentrate the liquid contained therein.

In case the liquid is not to be concentrated as far as or beyond the crystallizing-point, the lower part of the shell A may terminate in an attached liquid-reservoir, as shown at K, Fig. 2, having a discharge-valve *q* at the bottom, so that the liquid may be kept in constant circulation, being repumped and discharged through pipe C″, after passing through a heater or otherwise; but in either form of construction the liquid falling down the cylinder A always falls without contact with any obstruction into the liquid in the reservoir below the cylinder.

Suitable doors may be provided in the shell A for inspection and cleaning or repairing.

The shell A may be supported in place in any suitable manner, as by timbers, (shown at S S,) and may be enlarged, as shown in Fig. 2, at a point opposite the distributing pipe or chamber C′, to obtain a greater area of uptake at this point, and it may also be reduced higher up above the cylinder B, as shown in Fig. 2. Several of these devices may be placed alongside of one another in battery and may all be supplied by one blower for air, one pump for liquid, and one pan or one vessel D.

I do not confine myself to the precise construction of parts as herein shown, for these constructions may be varied in many ways to produce like results without departing from the spirit of my invention—as, for instance, as shown in Fig. 3, the sides of the shell A may be provided with perforations X X near the bottom thereof, and the air or gases be forced through them from an annular drum L, that is secured about the lower portion of said shell, and has an inlet-pipe *l* to make contact with the falling finely-divided liquid, or the air and gases so admitted may be exhausted upward by a fan or other suitable device applied to the upper part of the cylinder A, in which case the cylinder B and its direct attachments may be dispensed with and the distributing-pipe C be enlarged, as shown in Fig. 3; and in Fig. 3 the cylinder of shell A is, as a further development of the enlargement $A' A^2$, (shown in Fig. 1,) made to expand from a point a little above the distributing pipe or chamber C′ to the bottom, in order to prevent, absolutely, the contact of the falling liquid with the inner surface of the said cylinder; also, in Fig. 3, an exhaust-fan M, provided with shaft and pulley *r r′*, respectively, is fixed in a side of the shell A, above the distributing-pipe C, to draw the air or gases through the perforations *x x* to bring them in contact with the falling liquid and then eject them from the said cylinder. It will be seen that in this my improved apparatus there is no impediment, no object for the liquid to make contact with and become incrusted upon in falling through the cylinder, but that it falls directly into the liquid in the bottom receiver.

Having thus described my invention, I claim as new and desire to secure by Letters Patent—

1. An apparatus for vaporizing liquids, constructed substantially as herein shown and described, consisting of outer shell A, open at top and bottom, shell B, closed at top and bottom, having openings or perforations in its sides and centrally fixed within the shell A, so that an annular chamber is formed between them, liquid discharging and distributing chamber or pipe C′, with conduit C attached, conduit B′, connected with shell B, for introducing air or gases therein, and receiver D, set beneath shells A and B, all arranged substantially as herein shown and described.

2. In an apparatus for concentrating or evaporating liquids, the combination of an upright outer shell or cylinder expanding toward the bottom, provided with openings for the admission of air or gases, with an annular interior liquid-distributing chamber or pipe, an annular baffle-plate fixed on or about on a level with said pipe, and a fan for creating an upward current of air or gases within said cylinder, substantially as and for the purpose set forth.

3. In an apparatus for concentrating or evaporating liquids, the combination, with an upright outer shell or cylinder having an opening at the top and expanded at the bottom, and provided with an interior and concentric annular liquid-distributing chamber or pipe, and a central perforated air-distributing cylinder, of a liquid-receiver of a greater diameter than the lower end of said cylinder, fixed axially immediately beneath the same, substantially as herein shown and described.

In testimony that I claim the foregoing I have hereunto set my hand, in the presence of two witnesses, this 24th day of October, 1888.

MAURICIO M. MONSANTO.

Witnesses:

JACOB J. STORER,

CHAS. H. LOTT.

Plaintiff's Exhibit 2.

Patent No. 441,106.

(See Opposite ☛)

A. G. ENEAS.

SPRAY NOZZLE AND METHOD OF DISTRIBUTING LIQUIDS.

APPLICATION FILED DEC. 27, 1909.

1,101,264. Patented June 23, 1914.

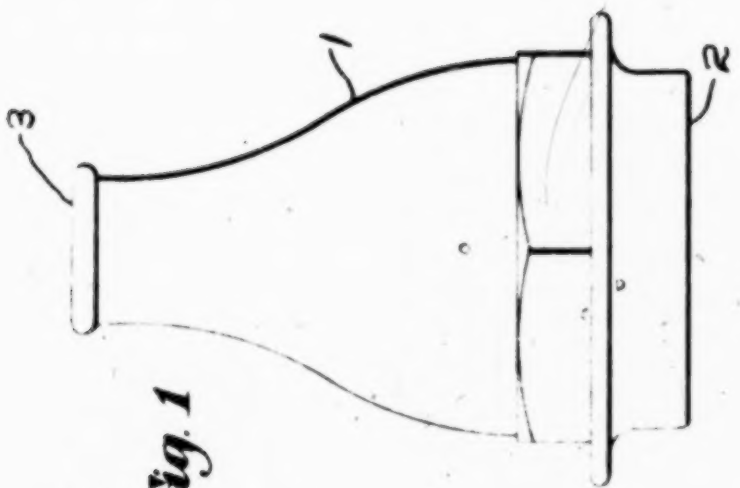


Fig. 1

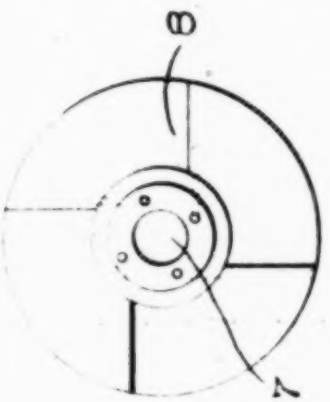


Fig. 2

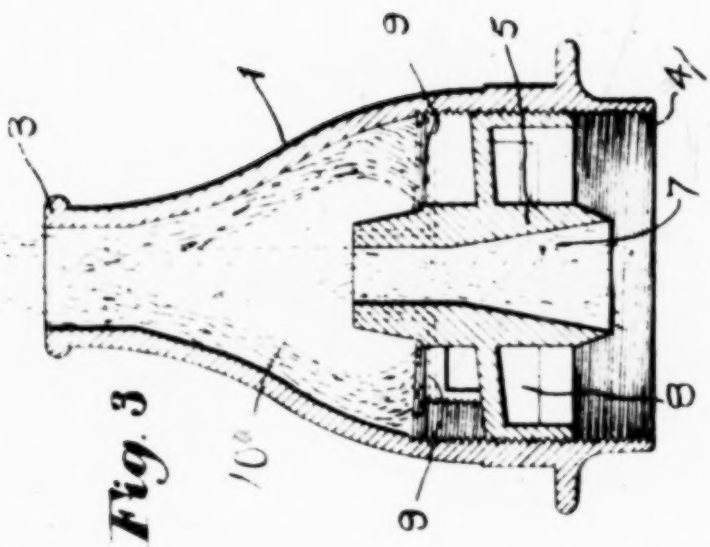


Fig. 3

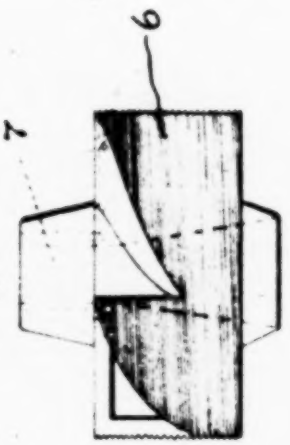


Fig. 4

Plaintiff's Exhibit 2.

Patent No. 1,101,264.

(See Opposite ☛)

Witnesses:
Earl L. Choate.
Ernest A. Telfer

Inventor:
Aubrey G. Encas,
by Emery & Booth.

Attys.

P 373 (2)

UNITED STATES PATENT OFFICE.

AUBREY G. ENEAS, OF WINTHROP, MASSACHUSETTS, ASSIGNOR TO SPRAY ENGINEERING COMPANY, OF KITTERY, MAINE, A CORPORATION OF MAINE.

SPRAY-NOZZLE AND METHOD OF DISTRIBUTING LIQUIDS.

1,101,264. Specification of Letters Patent. Patented June 23, 1914.

Application filed December 27, 1909. Serial No. 535,129.

To all whom it may concern:

Be it known that I, AUBREY G. ENEAS, a subject of the King of Great Britain, and a resident of Winthrop, in the county of Suffolk and State of Massachusetts, have invented an Improvement in Spray-Nozzles and Methods of Distributing Liquids, of which the following description, in connection with the accompanying drawings, is a specification, like letters on the drawings representing like parts.

This invention relates to fluid spray nozzles of general application, and to methods of distributing fluids.

In order that the principle of the invention may readily be understood, I have in the accompanying drawings disclosed a single type of nozzle embodying that part of my invention, and shall proceed to describe the best mode or manner known to me for practising such method.

In said drawings:

Figure 1 is a side elevation of a spray nozzle constructed in accordance with my invention and by which the method of my invention may be practised. Fig. 2 is a plan view of the inner liquid guiding member of the nozzle. Fig. 3 is a central longitudinal section taken through the inner and outer chambers of the nozzle; and Fig. 4 is a side elevation of the inner liquid guide member.

By the nozzle herein disclosed and in accordance with and in carrying out the method of my invention, I form and distribute over preferably an extended area, a spray composed of or comprising water or other liquid or suitable fluid, and which is, in the most effective embodiment thereof, of substantially uniform homogeneity throughout. Such spray is capable of use in many and varied relations. Among other uses, to which I am in no wise limited in the practice of my invention, I may enumerate the following:—For aerating municipal and other water supplies in reservoirs and other places; in connection with cooling ponds to spray the water heated in condensers of turbine or other engines; for spraying effluent in connection with sprinkling filter beds; for precipitating coal dust; for air washing; for condensing vapors; for humidifying air in chemical plants and in cotton mills, for oil burners, fire extinguishers, and in fact for many other uses not herein necessary to enumerate.

In carrying out my method, I project the fluid, of whatever nature, in a flaring spray, and in practising the method I preferably simultaneously produce both rotary and non-rotary jets, and mix, mingle or blend said jets within a confined space from which I cause the mixed, mingled or blended mass to issue in a spray of substantially uniform homogeneity.

Preferably the non-rotary jet is a central or axial one, and the rotary jet or jets surround the same and are caused transversely to impinge upon the axial or central jet with the result that said jets are sufficiently mixed to produce a spray of the character described when discharged from the confined space. In carrying out my method, I preferably provide a chamber in advance of the jet producing means, the said chamber being so shaped and of such volume as to permit an effective mixture or blending of the jets before they issue in spray form from said chamber. This result may be effected by so shaping the chamber or so positioning an abutment therein as to cause impact thereon of one or more of the surrounding jets and rebound thereof, with the result of a mixing or blending of these jets within the confined space.

In that embodiment of the nozzle of my invention herein selected for illustration and by which I may practise my invention, the nozzle comprises a shell or casing 1 constituting an outer mixing chamber and having an entrance 2 for the water or other liquid to be sprayed and a discharge orifice 3. The nozzle may be suitably connected to a hose or other means for conveying the fluid or liquid to be sprayed. For the purpose, I have herein represented the nozzle 1 as interiorly threaded at 4. Within the shell 1, I form or position an inner liquid guiding member 6 herein represented as detachable from the shell 1 and as exteriorly threaded at 4, so that it may be screwed into position in the shell 1, forming the mixing chamber 10. The inner member 6 may be constructed in any suitable manner to produce the desired results. In order to effect the spraying referred to, I so construct the inner liquid guiding member that it will

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discharge or direct into the mixing chamber 10 a pluraiity of jets as hereinafter set forth. For this purpose, I may employ a single central jet and one or more exterior jets which, if a plurality thereof be employed, surround the central jet. Preferably at least one of these divergent jets is a whirling one, the jet being given a rotating or whirling character in any suitable manner. Within the scope of my invention, the central jet may be a whirling jet or the surrounding jet or jets may be of that character.

Obviously, the method is not dependent upon the particular form of inner guiding member 6, or the means for positioning or forming the same within the shell or casing.

In that form of nozzle shown in Figs. 1 to 4 inclusive, the central jet is produced by the central passage 7 formed in the liquid guiding member 6 and receiving the water or other liquid or fluid directed thereinto under pressure and from any suitable source. Surrounding the central straight passage 7 are a plurality of divergent curved passages 8 formed between the wall of the central passage 7 and the exterior wall of the member 6. These passages 8 likewise receive liquid or fluid under pressure from the same source and impart a whirling or rotating action to the jets passing therethrough as well as a centrifugal action. The jets passing through the passages 7 and 8 are primarily of equal velocity, but the velocity of the jets passing through the passages 8 is reduced below that of the central jet, owing to the contact of said surrounding jets with the inner wall of the mixing chamber 10. The jets passing through said passages 7 and 8 converge and meet within the mixing chamber 10, the tapering formation of this chamber 10 compelling the mixing of said jets. The impinging of the jets upon each other thoroughly breaks them up and compels a thorough mixing and distribution of the constituent parts thereof, so that as the liquid emerges from the nozzle it spreads into a cone whose apex is the orifice of the nozzle, the spray being substantially uniformly and thoroughly distributed throughout the entire cone.

I have, in the selected form of nozzle, shown the chamber as having vanes shaped to produce rotating or centrifugal jets, said vanes in such selected form of nozzle each extending less than half way about said chamber, and being curved so as gradually to decrease the pitch of the vanes in the direction of flow of the jets and thus gradually to increase the tangential velocity of said jets past the vanes. I have herein shown said vanes as being substantially quadrantal in shape, it being clearly evident that this construction may be widely varied within the scope of my invention.

In other spraying nozzles with which I am familiar the spray is discharged in a cone, but the spray is substantially confined to elements of the cone, so that in reality the spray emerges in a sheet of conical form, it also being common to provide a central jet discharged unmixed with said conical sheet and intended for ornamental effect merely. My nozzle and the method herein disclosed on the contrary produce a spray so uniformly distributed that were sections taken through the cone to which I have referred, they would exhibit a substantially uniform distribution of the spray throughout such section. This uniform distribution is owing to the mixing compelled by the jets 7 and 8 and the distribution in all directions resulting therefrom. I am aware that whirling members have been positioned within nozzles and provided with passages for the liquid to be sprayed, but the result of such construction is to impart merely a centrifugal action to the spray so that in effect merely a shell or conical sheet is formed.

If desired, I may provide means for introducing air into the mixing chamber 10 in order to compel a more thorough intermingling of the jets. While this may be done in any suitable manner, I have herein represented air passages 9 formed in the walls of the member 6 and admitting external air into the interior of the mixing chamber 10. Each air passage 9 is preferably of the general angular character shown so as to introduce the air along radial lines and to discharge the same substantially axially and at points surrounding the central jet issuing through the passage 7.

It will be apparent that within the mixing chamber 10 a partial vacuum is formed between the jets issuing from the passages 7 and 8. Owing to the formation of this partial vacuum, the external air is sucked in through the passages 9, and although entering at atmospheric pressure it has greater pressure imparted thereto by the jets issuing through the passages 7 and . It is evident that the external air may be admitted, if at all in any suitable manner and that within the scope of my invention air primarily under greater than atmospheric pressure may be utilized.

It will be apparent that the air when employed in any form of my invention tends further to atomize the fluid at its exit from the nozzle by reason of the expansion of the air after its discharge from the nozzle.

The mixing chamber is relatively large compared to the orifice through which the fluid issues from the nozzle. Said chamber is of sufficient volume and so shaped as to permit and effect blending or mixing of the jets before issuance from the nozzle. In that form of nozzle herein illustrated, the cham-

ber is so shaped as to direct the rotating or centrifugal jet or jets across the path of the axial jet within the chamber, before the jets issue from the nozzle, thereby to cause said rotating jet or jets to be sprayed by said axial jet, so as to increase the spraying angle and form a full spray. In that form of nozzle herein shown, the cross sectional area of the mixing chamber exceeds the combined cross sectional area of the axial and surrounding jets. The chamber is in advance of the means for producing the jets and has its exit spaced sufficiently therefrom to permit a blending of the jets within the nozzle, the chamber being shown as having an impact-receiving formation to compel said blending. The said chamber is shaped to compel transverse impingement upon and ensuing blending of the rotary jet or jets with the non-rotary jet. The rotary jet or jets is or are transversely projected into blending union with the non-rotary jet, before issuance of the jets from the nozzle, it being evident that I provide an impact-receiving and rebound-compelling abutment positioned in advance of the jet-producing means to cause blending of the jets before issuance from the nozzle.

It will be apparent from the foregoing description that the nozzle herein disclosed is of great simplicity of construction and is of much capacity. By reason of the uniform distribution of the spray, there is a very substantial freedom from all drift. Moreover by reason of the fact that the nozzle contains no moving parts, it is more durable and is of cheaper construction.

It will also be apparent that in accordance with my method, the liquid or fluid of the jets is mixed or blended within a confined chamber or space, and that the mixed mass issues therefrom in a spray which is of substantially uniform homogeneity throughout.

It will furthermore be apparent that the liquid or fluid of the jets is sprayed and atomized and is so thoroughly distributed that all points within the range of the nozzle receive substantially an equal amount of sprayed or atomized liquid or fluid.

Having thus described one illustrative embodiment of the nozzle of my invention and one manner of practising my method, I desire it to be understood that although specific terms are employed, they are used in a generic and descriptive sense and not for purposes of limitation, the scope of the invention being set forth in the following claims.

I claim—

1. In a nozzle, the combination of means for producing a non-rotating axial jet, means for producing one or more surrounding rotating or centrifugal jets, and a relatively large mixing chamber in advance of the means for producing the axial jet, having guiding means positioned to direct said centrifugal jet or jets across the path of the axial jet within said mixing chamber before said jets issue from the nozzle, whereby said jets are mixed.

2. A spray nozzle comprising in combination an inner fluid directing member and an outer member inclosing a vacuum and fluid mixing chamber, said members arranged to produce a partial vacuum automatically within the nozzle, and passages for leading the air from the outside of the nozzle to the vacuum chamber, for automatically mixing the air with the fluid under pressure before emission from the nozzle.

3. A fluid spray nozzle comprising in combination a chamber having an emission orifice and wherein a vacuum is automatically formed by the fluid in its passage through the nozzle, means for producing a plurality of jets of different directions of flow through said nozzle, one or more air passages admitting external air to the vacuum chamber, said chamber being so formed as automatically to mix the air and said jets in the nozzle before their emission therefrom.

4. A fluid spray nozzle comprising in combination a chamber having an emission orifice and wherein a vacuum is automatically formed by the fluid in its passage through the nozzle, means to produce a whirling jet within the nozzle, one or more air passages for admitting external air to said vacuum chamber, said chamber being so formed as automatically to mix the admitted air with said whirling jet before its emission from the nozzle.

5. In a nozzle, the combination of means for producing a non-rotating axial jet, means for producing one or more surrounding rotating or centrifugal jets, and a chamber in advance of the means for producing said jets and having converging walls, shaped to direct said rotating jet or jets across the path of the axial jet within the said chamber before the jets issue from the nozzle.

6. In a nozzle, the combination of means for producing a non-rotating axial jet, means for producing one or more surrounding rotating or centrifugal jets, and a chamber in advance of the means for producing said jets, and having guiding means shaped to direct said rotating jet or jets across the path of the axial jet within said chamber before the jets issue from the nozzle, thereby to cause said rotating jet or jets to be spread by said axial jet, so as to increase the spraying angle and form a full spray.

7. A spray nozzle comprising in combination, means for producing an axial, non-rotating jet and one or more centrifugal surrounding jets, and a surrounding mixing chamber beyond the jet orifices, in which all

the jets are mixed before emission from the nozzle, the cross sectional area of said mixing chamber exceeding the combined cross sectional area of said axial and surrounding jets.

8. A spraying nozzle comprising in combination, a chamber having vanes shaped to produce rotating or centrifugal jets, said vanes each extending less than half-way about said chamber, and being curved so as gradually to decrease the pitch of the vanes in the direction of flow of the jets and thus gradually to increase the tangential velocity of said jets past the vanes.

9. A spraying nozzle comprising in combination a chamber, having vanes shaped to produce rotating or centrifugal jets, said vanes each being substantially quadrantal in shape and being curved so as gradually to decrease the pitch of the vanes in the direction of flow of the jets and thus gradually to increase the tangential velocity of said jets past the vanes.

10. In a nozzle, means for simultaneously producing both rotary and non-rotary jets and means for mingling said jets before issuance from the nozzle.

11. In a nozzle, means for simultaneously producing both a rotary and a non-rotary jet and means transversely to project said rotary jet into blending union with said non-rotary jet, before issuance of said jets from the nozzle.

12. In a nozzle, in combination, means for producing a non-rotary axial jet, means for producing one or more surrounding rotary jets, and a chamber in advance of said means, shaped to compel transverse impingement upon and ensuing blending of said rotary jet or jets with said non-rotary jet.

13. In a nozzle, in combination, means for producing a non-rotary jet and one or more rotary jets, and an impact-receiving and rebound compelling abutment positioned in advance of said means to cause blending of said jets before issuance from the nozzle.

14. In a nozzle, in combination, means for producing a non-rotary jet and one or more rotary jets, and a chamber in advance of said means and having its exit spaced sufficiently therefrom to permit a blending of said jets within the nozzle, said chamber having an impact receiving formation to compel said blending.

15. In a nozzle, in combination, means for producing a non-rotary jet and one or more rotary jets, and a chamber in advance of said means and of sufficient volume and shaped to permit and effect blending of said jets before issuance from the nozzle.

16. That method of distributing liquid over an extended area which consists in simultaneously producing both rotary and non-rotary jets and in causing a mixture of said jets within a confined space.

17. That method of distributing liquid over an extended area which consists in simultaneously producing both rotary and non-rotary jets and in causing the mixture of said jets within a confined space and the issuance thereof from said space in a spray of uniform homogeneity.

18. That method of distributing liquid over an extended area which consists in simultaneously producing both a rotary and a non-rotary jet, and in transversely projecting the rotary jet in a confined space into blending union with said non-rotary jet and in thereafter effecting issuance of said blended jets from said space as a spray.

In testimony whereof, I have signed my name to this specification, in the presence of two subscribing witnesses.

AUBREY G. ENEAS.

Witnesses:
IRVING U. TOWNSEND,
F. IRENE CHANDLER.

Plaintiff's Exhibit 2.

Patent No. 2,375,922.

(See Opposite ☛)

Note

Page 575-1 is on card 6

Patented May 15, 1945

2,375,922

UNITED STATES PATENT OFFICE

2,375,922

TREATMENT OF SOLUTIONS TO OBTAIN SOLID MATTER THEREFROM

Finn Jeremiassen, Oslo, Norway; vested in the Alien Property Custodian

Application September 15, 1939, Serial No. 295,147
In Norway October 5, 1938

5 Claims. (Cl. 23—295)

This invention relates to the treatment of solutions to obtain solid matter therefrom and has for its object a process as well as an apparatus by means of which it is made possible to obtain the separated solid matter in the form of comparatively coarse granules. According to the invention the required increase in the concentration of the treated solution is brought about by contacting the solution with a gas.

It is known that it is possible to increase the concentration of a solution by causing a gas to act on same, by evaporation, cooling or by chemical reaction, for example by the action of a gas containing ammonia, on a liquid containing sulphuric acid, whereby sulphate of ammonia is formed. The means hitherto employed to obtain a large area of contact between liquid and gas have been either to force the gas down into the solution and allowing it to ascend in the form of bubbles, or also to cause the liquid to pass through scrubbers, in the form of cascades or as a rain of jets or drops, downwards through a flowing gas.

When it is desired, however, to carry the concentration so far as to a state of supersaturation, such as is necessary to bring about precipitation of dissolved solids, the means hitherto employed have been found to be practically in applicable because after a short time of operation obstructing coatings are formed on parts of the solid bodies contacting with the supersaturated solution in the gas space.

In the specification of a prior U. S. A. Patent No. 1,478,337 I have mentioned the various advantages which are attainable by operating with degrees of saturation within the metastable limit particularly, when it is desirable to obtain products in the form of coarse granules.

Coarse granular precipitates frequently present several advantages such as a lower cost of centrifugal and drying treatment, higher degree of purity (owing to the fact that the percentage of adhering impure solution is less), decreased formation of dust, decreased agglomerating and caking tendency during storage etc.

In spite of the disclosures in the mentioned U. S. A. patent specification No. 1,478,337 the problem of using a gas to bring about supersaturation in crystalizers has not until now been solved in a satisfactory manner in connection with the manufacture of comparatively large-sized crystals.

Primarily this is due to the difficulties connected with the establishment of a uniform supersaturation, which at no point shall exceed the rather narrow limits of metastable supersaturation. If the flow of liquid through the gas space is slower at some places than at others, parts of the liquid may from this reason happen to be subjected to the supersaturating effect of the gas for too long a time. Most markedly this will take place in connection with splashes causing drops of liquid to adhere to solid bodies in the path of the flowing gas resulting in the formation of finely granular salt and coatings.

I have observed that the main difficulties are due to these circumstances and the present invention is based on this observation.

An important characteristic feature of the process, which is the object of the invention, consists therein that the solution to be concentrated by contact with a gas is introduced into the gas space in an approximately saturated condition and by the action of the gas is caused to be supersaturated to a point within the metastable limits. The supersaturation so produced is thereupon released by contact with crystals of the same substance as that which it is desired to precipitate, whereupon the solution is again returned to the gas space to be again subjected to concentration to a point within the metastable limit of supersaturation.

In the following the invention is described with reference to the accompanying drawing, in which Figs. 1 and 2 diagrammatically illustrate by way of example two types of apparatus adapted to carry the invention into effect.

Referring to Fig. 1 the numeral 1 designates a pump forcing approximately saturated solution up into a container 2, having a bottom provided with holes. The liquid passing through said holes flows freely through the space 4 below said container (2). Through a pipe 5, an annular chamber 6 and openings in a jacket wall 7 a hot gas is forced into the space 4. This brings about evaporation from the surface of the liquid streams or jets which to a greater or less degree will be disrupted so as to form drops. This results in the liquid becoming supersaturated to within the metastable limit, viz. to a degree of supersaturation at which no crystals are formed.

The cooled gas flows out through the pipe 8 as indicated by arrows, while the supersaturated solution collects in the hopper 9, from which it returns to pump 1 through pipe 10 and crystal suspension 11.

In continuous operation fresh solution is constantly supplied to replace evaporated liquid and deposited solids taken up on the crystals in the suspension 11. When this added solution is un-

dersaturated, it may be used to utilise the remaining heat (or chemically active constituents, such as for example ammonia) in the escaping gas by passing the added fresh solution in the most intimate contact possible with the escaping residual gas as diagrammatically illustrated in the drawing. The solution in this example is passed through a pipe 12 down into a cup 13, with holes in the wall or with overflow edge. From this cup the liquid flows downward in such a way that the escaping residual gas from pipe 8 must pass through the "veil" of liquid.

In the case of the volume of added solution being so small that it will be difficult to bring about a uniform distribution of the "veil" over the entire cross sectional area of the container, its volume may be increased by first mixing it with a part of the circulating liquid from the apparatus.

Instead of causing the added undersaturated liquid to fall freely downward, the liquid may also be brought to trickle in cascades or through a scrubber in which the residual gases ascend.

The crystals in the suspension 11 grow as a result of the supersaturation being released on them. The larger ones sink farthest downwards and are continuously or intermittently removed through pipe 14.

In the operation of the described plant no part of the liquid will be subjected to the supersaturating effect of the gas for a too long time, and there are no solid bodies in the path of the particles of liquid, onto which liquid might adhere to form coatings. The only solid body with which the particles of liquid enter into contact while in the flowing gas, viz. the bottom 3, is left by the liquid before there has been time enough to arise any substantial degree of supersaturation. Further the re-supersaturation of the liquid at each cycle is released in the crystal suspension 11, and this means that the total supersaturation at no point will arise above the metastable limit, so that the holes in the bottom 3 will not be obstructed. The fact that the gas flows inwards into space 4 further counteracts splashing from the streams of liquid on to the jacket wall 7 particularly in the case of this latter having been given a tapering form as illustrated, because the spreading or disintegration of the streams or jets into drops will increase the longer the streams have moved downward.

It is also possible to dispense with the jacket 7 because also in this case it is possible to obtain sufficent relative velocities between gas and liquid when there is provided for the production of a strong whirling motion in the gas already before it enters between the streams of liquid.

The process as explained operates with degrees of supersaturations, which are at all points within the metastable limit so that coatings (crusts) on the parts of the apparatus are avoided. This means that the supersaturation per cycle which shall give the entire production, is quite small, usually only a fraction of a percent of the entire contents of salts in the liquid. For this reason very large quantities of circulation must be employed, viz. many times as large as the quantity of liquid added per second through 12. This is an important new feature of the present process, inasmuch as it is only thereby rendered possible to employ a great number of liquid jets in the space 4, i. e. a very large contacting surface between the liquid and the gas without having to resort to the use of spray, or quite thin films of liquid as employed in scrubbers, as this would result in formation of fine salt and coatings caused by the limit of the metastable supersaturation being locally exceeded, as explained above.

The method of causing liquid to fall freely down through the space through which gas is flowing, viz. without solid bodies having at any point a chance to produce injurious local non-uniform motion, is also capable of being carried into effect in other ways than illustrated in Fig. 1.

In Fig. 2 is shown as an example a modified embodiment of the invention. In this case the circulation pump 1 forces the circulating liquid through a pipe 17, provided with holes and into the space 4, through which gas is flowing.

If liquid splashes on the wall 15 of chamber 4 and this splashing is insufficient to maintain the wall moist, the undesirable effects of the splashes can be avoided by constantly causing an auxiliary flow of liquid to wash over the wall 15, so that no part of the wall is alternately dry and wet (which would result in the formation of coatings on the wall). To bring about such continuous washing of the wall one may for example make use of a secondary pipe 16, and an annular slot 18. The main principle on which the present invention is based implies that the circulating quantities of solution, as above explained, are very large, and from this reason the employed "washing" flow of solution does not involve any appreciable decrease in the quantity of solution which is utilized as a shower of streamlets or jets.

Fresh solution is introduced through pipe 19 or for example through 16.

In order to secure a uniform distribution of the gas blown in through pipe 5 and also to obtain comparatively large gas velocities in an inward radial direction in space 4, so that jets of solution from pipe 7 shall not splash on to the walls of the chamber, an annular shield 20 may also be arranged.

It is not necessary to maintain atmospherical pressure in the chamber 4. This space may also be kept under vacuum.

As an example of fields in which the present invention can be adapted may be mentioned—in addition to the manufacture of ammonium salt above described—the crystallization of anhydrous sodium sulphate by forcing hot combustion gases through space 4, so as to cause evaporation of a part of the water in each cycle of the flowing solution.

I claim:

1. Apparatus for treating solutions to precipitate soluble matter therefrom comprising a gas chamber, a liquid distributing device at the top of said gas chamber and so arranged as to cause a shower of liquid to fall freely through the gas chamber, a gas supply jacket surrounding the said gas space chamber, the wall between the jacket and the gas chamber having holes to allow gas from the jacket space to pass into the said gas chamber as inwardly directed streams, an exit opening for residual gas being arranged at a central point in relation to the liquid distributing device means for passing a gas into said supply jacket, through said holes and through said gas chamber to said exit, said gas being adapted to produce supersaturation of said liquid within the metastable limit, a crystallizing chamber, means for passing said supersaturated liquid through said crystallizing chamber and means for recycling the liquid from said

crystallizing chamber to said liquid distributing device.

2. A crystallizing apparatus comprising a crystallizing chamber and a superimposed gas chamber, disturbing means for causing jets of a solution of a material to be crystallized to pass through said gas chamber, means for collecting the gasified solution and for passing it into said crystallization chamber, a gas supply jacket surrounding said gas chamber and separated therefrom by a wall, said wall being provided with perforations adapted to permit gas from the jacket to pass into the gas chamber in an inwardly directed stream, said wall having the form of an upwardly tapering cone.

3. A crystallizing process which comprises passing a substantially saturated salt solution containing a reagent in a freely falling shower through an open, unobstructed zone in such manner that the shower of solution is surrounded by a gas space, passing a gas inwardly from said gas space into said zone and upwardly in counter-current to said shower, said gas being reactive with the reagent in solution to produce a supersaturated solution of a salt, then passing said supersaturated solution thus produced upwardly through a suspension of crystals of the salt so produced, thereby releasing the supersaturation of said solution and causing crystallization of said salt on said suspended crystals, separating the resulting crystals from the solution and recycling the latter to said shower.

4. A crystallizing process which comprises passing a substantially saturated solution of a crystalline compound in a freely falling shower through an open, unobstructed concentrating zone in such manner that the shower of solution is surrounded by a gas space, passing a gas inwardly from said gas space into said zone and upwardly in counter-current to said shower, said gas being adapted to produce an increase in concentration of said solution to produce supersaturation thereof, then passing said supersaturated solution upwardly through a suspension of crystals of said compound of a size such that the crystals tend to fall through said solution by gravity thereby releasing said supersaturation and causing crystallization of said compound on said crystals, separating the resulting crystals from the solution and recycling the latter to said shower.

5. Apparatus for treating saturated solutions to precipitate crystallizable matter therefrom comprising an open concentrating chamber, a solution distributing device at the top of said chamber having a plurailty of openings in the bottom thereof for the passage of the solution therethrough in a shower to fall freely through the chamber, a gas supply comprising a jacket having a wall surrounding the said chamber, the wall between the jacket and the chamber having openings adapted to allow the gas to pass from the jacket into the said gas chamber through said shower as inwardly directed streams the wall being so positioned with respect to the said solution distributing device that the shower of falling solution does not contact said wall, a crystallizing chamber for containing a suspension of crystals of said crystallizable matter therein and means including a pump for passing said supersaturated solution through said suspension whereby crystallization of said solution on said suspended crystals takes place.

FINN JEREMIASSEN.

CERTIFICATE OF CORRECTION.

Patent No. 2,375,922. May 15, 1945.

FINN JEREMIASSEN.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, second column, line 40, for "pipe 7" read --pipe 17--; page 3, first column, line 5, claim 2, for "disturbing" read --distributing--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 21st day of August, A. D. 1945.

Leslie Frazer

(Seal) First Assistant Commissioner of Patents.

Plaintiff's Exhibit 2.

Wethly Article.

(See Opposite ☛)

Note

Page 377 is on card 6

Plaintiff's Exhibit 2.

Spraco Catalog Sheets.

(See Opposite ☛)

Note

Page 379 is on card 10

Plaintiff's Exhibit 3.

(See Opposite ☛)

Note

Page 381 (sheets 1 - 3) are on card 10
Page 381 (sheets 4 - 10) follow this frame

Patented July 8, 1947 2,423,794

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UNITED STATES PATENT OFFICE

2,423,794

PRODUCTION OF AMMONIUM SULPHATE

Carl Otto, Manhasset, N. Y., assignor to Fuel Refining Corporation, New York, N. Y., a corporation of Delaware

Application August 25, 1943, Serial No. 499,986

3 Claims. (Cl. 23—119)

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The general object of the present invention is to provide an improved method of and improved apparatus for converting the ammonia content of distillation gas, and particularly coke oven gas, into ammonium sulphate by passing the gas into a so-called saturator containing a bath of weak sulphuric acid with which the gas is brought into scrubbing contact so that the ammonia elements in the gas and the sulphuric acid elements in the bath combine to form sulphate of ammonia. The later subsequently crystallizes out of the bath liquor. Ordinarily, the sulphate crystals thus formed, accumulate in the lower portion of the bath and are withdrawn from the saturator in a carrying stream of bath liquor by a discharge pump or ejector.

Various forms of saturators of the general character described have long been, and now are in general use in this country usually by product coke oven plants. As a result of plant expansions, and economies to be obtained by their use, there is now a substantial need for saturator units of larger individual operative capacity than has been customary heretofore.

A major object of the present invention is to provide a novel method of and means for bringing the gas into scrubbing contact with the bath liquor which are efficient and well adapted for practical use in saturator units of larger capacity than have been heretofore customary. The invention is not restricted to use in and in connection with saturators originally devised and adapted for such use, as existing saturators of various types may be made more efficient and may have their operative capacities greatly increased by relatively inexpensive modifications which adapt them for use in accordance with the present invention.

Another major object of the invention is to effect adequate and efficient scrubbing contact of the gas and bath liquor with a substantially smaller expenditure of potential energy, or gas pressure drop, in the saturator than is required in the normal operation of saturators heretofore in use. The reduction in the saturator gas pressure drop made possible by the use of the present invention results in a worthwhile saving of gas pumping energy and expense.

The present invention is primarily characterized by the movement of the gas in a generally horizontal direction through a scrubbing zone gas space maintained between an upper wall surface and a lower bath liquor surface depressed by the pressure of the flowing gas. At the outlet end of said gas space its roof is several inches

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below the upper surface of the bath liquor surrounding the scrubbing zone and into which the gas passes from the gas space. The latter is of sufficient extent in the direction of gas flow and is sufficiently restricted in the transverse direction, to maintain liquor and gas flow conditions which result in the conversion into ammonia sulphate of much of the ammonia content of the gas while the latter is passing through said gas space. I believe that the demonstrated practical merit of the invention is due in substantial part, at least, to the fact that the stream of gas flowing rapidly through the relatively shallow horizontal gas space, subjects the liquid forming the bottom wall of said space to ripple creating and atomizing actions. The local circulation of the bath liquid incident to the formation of ripples and the dispersion of the atomized liquid in the gas stream, each contribute to the rapid conversion of the free ammonia elements of the gas into ammonium sulphate by providing opportunity for contact of the ammonium elements in the gas with bath liquor acid elements not previously converted into ammonium sulphate.

While it is essential to the full attainment of the advantages of the invention that the direction of gas flow through the free gas space of the scrubbing zone should be approximately horizontal, I have found it practically desirable to incline the wall surface forming the roof of said gas space at a small angle to the horizontal with the lower edge of said wall surface at the gas discharge edge of said space. Said angle which may vary from a minimum of four or five degrees to a maximum not greater than fifteen degrees. Unless the wall forming the roof of the free gas space is so inclined, gas flow conditions and bath liquor surging actions may occur which will materially interfere with the efficiency of the scrubbing action, and which tend to cause objectionable deposits of sulphate of ammonia crystal or "salt," on said wall.

In a preferred practical form of the present invention, the roof wall of the scrubbing zone gas space is formed by the lip portion of an outwardly flared, downwardly opening mouthpiece of a "cracker pipe" through which the gas is passed downward into contact with the bath liquor into which said mouthpiece extends, but said roof wall may take other forms.

The present invention may be used with substantial advantage in saturators operated to produce sulphate crystals of the relatively small size usually produced heretofore, and may be

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used with even greater advantage in saturators operated to produce so-called "large" ammonium sulphate crystals. One well known grade of such "large" crystals comprise crystals of which 40% are too large to pass a 35 mesh screen and only 5% are small enough to pass a 70 mesh screen; whereas of the small crystal sulphate constituting the bulk of the ammonium sulphate heretofore produced in saturators, 80% or more will pass through a 35 mesh screen and about 40% will pass through a 70 mesh screen.

The various features of novelty which characterize my invention are pointed out with particularity in the claims annexed to and forming a part of this specification. For a better understanding of the invention however, its advantages and specific objectives attained with its use reference should be had to the accompanying drawings and descriptive matter in which I have illustrated and described the preferred embodiments of the invention.

Fig. 1 is an elevation partly broken away and in section;

Fig. 2 is a plan view of the saturator shown in Fig. 1;

Fig. 2A is a section on the line 2A—2A of Fig. 2;

Fig. 3 is a plan view of a portion of the cracker pipe mouthpiece shown in Figs. 1 and 4;

Fig. 4 is a reproduction on a larger scale of the cracker pipe mouthpiece section shown in Fig. 1;

Fig. 5 is a plan view of a portion of a modified form of the mouthpiece shown in Figs. 1–4;

Fig. 6 is a diagrammatic sectional elevation of a portion of a saturator differing in form from that shown in Fig. 1;

Fig. 7 is a diagrammatic sectional elevation of a portion of another modification of the saturator shown in Fig. 1;

Fig. 8 is a chart illustrating test apparatus results obtained with different rates of gas flow and with different roof inclinations;

Fig. 9 is a sectional plan of a third saturator form;

Fig. 10 is a partial section on the line 12—12 of Fig. 9;

Fig. 11 is a sectional plan of a fourth form of saturator; and,

Fig. 12 is a section on the line 14—14 of Fig. 11.

In Figs. 1–4, I have illustrated an ammonia saturator comprising a tank A which holds a body of bath liquor B in its lower portion and is of a type and form long in use in this country, and in which I have replaced the means originally employed to pass the gas into contact with the bath liquor by a vertically disposed cracker pipe C having a mouthpiece D at its lower end which is shaped and disposed in accordance with the principles of the present invention.

The tank A illustrated comprises a body portion which in horizontal cross section is in the form of an ellipse with flattened sides; a rounded upper portion A′ which may be described as an elongated dome; and a bottom portion which is formed with two depressions or wells A^2, each serving as a receiver for crystals settling to the bottom of the saturator. The upper tank portion A′ is formed at its top with two openings surrounded by external flanged pipe connections A^3 of the tank structure. The two flanged pipe connections A^3 are displaced in opposite directions from the vertical tank axis. The cracker pipe C has its upper end secured in the opening surrounded by one of the flanged pipe connections A^3 to which the gas supply pipe or main E is attached. The saturator gas discharge pipe F is secured to the second flanged pipe connection A^3 and receives gas from the saturator gas space surrounding the cracker pipe C. The cracker pipe mouthpiece D has its axis coincident with the vertical tank axis, the body portion of the cracker pipe being correspondingly inclined to said axis. To prevent movement of the lower end of the cracker pipe and its mouthpiece away from the tank axis under the action of gravity, the lower end of the cracker pipe is connected to the upper tank portion A by one or more suitably disposed stay bolts or tension rods C′.

The discharge pipe F passes gas into a so-called acid catcher G which collects bath liquor entrained in the gas passing out of the saturator through the pipe F, the gas passing away from the acid catcher G through piping not shown. Sulphate crystals and carrying bath liquor are withdrawn from the wells A^2 through separate suction pipes H. The suction pipes H pass out of the saturator through wall openings surrounded by external nozzle portions A^4 of the tank structure through which the pipes H are connected to pumping or exhausting apparatus (not shown) but serving in a known manner to pass the crystals and liquor mixture to the usual crystal and liquor separating apparatus from which bath liquor separated from the crystals may be returned to the saturator. As shown, a horizontal spray ring I located in each well A discharges upwardly directed jets of air or gas supplied under a suitable pressure through corresponding pipes I′ which extend through saturator wall openings surrounded by external nozzle portions A^5 through which the pipes I′ are connected to means supplying air or gas under suitable pressure. The jets discharged by the spray pipe rings I agitate the bath liquor and tend to retard the movement of very fine sulphate crystals to the inlet ends of the suction pipes H.

The body portion of the saturator is formed with an opening surrounded by an external nozzle A^6 through which bath liquor may overflow into an external tar separator J of known form. As shown, the saturator J comprises a tank in which there is a seal pot or chamber J′ closed at its lower end and open at its upper end, into which liquor overflowing from the saturator is passed through a vertical pipe J^2 having its open lower end adjacent the lower end of the seal pot J′ and having its upper end connected to an overflow box K secured to the end of the nozzle A^6. As shown, the overflow box K is in the form of a lead lined pipe fitting having one open side in register with the nozzle A^6. The member K has a lower nozzle end portion K′ which is mechanically connected to the upper end of the discharge pipe or spout J^2, and has an upper nozzle end portion closed by a head K^2. The latter is formed with a central passage through which a longitudinally adjustable rod or stem K^3 extends into connection with the upper end of a vertically disposed tubular sleeve or weir member K^4. The latter is fitted snugly in, and is longitudinally adjustable in the lower nozzle portion K′ of the member K. The level of the upper end of said sleeve K^4 corresponds to and determines the saturator bath liquor level B′, and the bath level B′ may be vertically adjusted by adjusting the rod K^3 relative to the head K^2. As shown, the rod K^3 may be secured in any of different positions by a cross pin K^5 extending through one or another of a series of transverse passages in the rod K^3.

The bath liquor discharged from the saturator through the nozzle A^6 and fitting K into the seal

pot J', overflows from the latter through a weir notch J^3 into the tank J. The tar separating from the gas passing through the saturator, floats at the top of the saturator bath and is carried out of the saturator by the bath liquor overflowing through the nozzle A^6 into the tank J from which the tar may be manually skimmed off or otherwise eliminated. A baffle J^4 prevents the tar entering the tank from reaching the liquor outlet J^5 of the tank. The continuous and relatively expeditious removal of tar from the saturator thus effected, contributes to the production of sulphate crystals much freer from tar contamination and tar discoloration than is possible when no such tar elimination is effected.

The bath liquor withdrawn from the saturator through the crystal discharge pipes H, and that carried into the acid catcher by the gas passing away from the saturator, and the bath liquor passing out of the saturator through the overflow opening surrounded by the nozzle A^6 in the tar separator J, may all be returned to the saturator. All or a substantial portion of such bath liquor may be returned to the saturator through pipes L extending into the saturator through wall openings surrounded by external nozzles A^7. As shown in Figs. 1 and 2, the discharge ends of the return pipes L extend in the same generally tangential relation to the vertical axis of the saturator so that the return liquor discharged by the pipes tends to maintain a bath circulation in the counter-clockwise direction about the vertical tank axis. This circulation contributes to a suitably rapid and complete combination of ammonia in the gas with the acid in the bath liquor and also contributes to a movement of the tar floating at the top of the bath, toward and into the overflow connection leading to the tar separator J.

As those skilled in the art will understand, all pipe and tank surfaces, etc. exposed to contact with the bath liquor should be formed of or provided with a protective coating of material such as lead or Monel metal which is immune to the corrosive action of the bath liquor.

To eliminate or minimize salt deposits, a washing liquid, ordinarily hot water, may be sprayed into the saturator through suitable spray pipes such as the spray pipe M entering the saturator through the upper tank part A', and spray pipes M^1 which enter the saturator tank through lateral openings surrounded by external nozzles A^8 and spray pipes M' may well serve to atomize the water which they spray into the saturator, all as described and claimed in my prior application for patent Serial No. 473,949, filed January 29, 1943. I have found, however, that in the operation of a saturator at the high capacity rate made possible by the use of the present invention, the relatively large amount of bath liquor splashed onto the saturator internal walls above the bath liquor level B' greatly eliminates or greatly reduces the liability of objectionable salt deposits on those walls.

Pipes MA extending into the saturator through the nozzles A^8 discharge a mixture of steam with air or gas and preferably coke oven gas, into the bath liquor adjacent the wall of the tank and a small distance below the bath liquor surface B'. As shown the discharge ends of the pipes MA are so shaped that steam and gas jets into the bath liquor alongside the tank wall and are so directed that they tend to maintain a bath liquor circulation about the vertical axis of the tank. The steam in the steam-air or steam gas mixture discharged into the bath liquor through the pipes MA locally heats the bath liquor at the top of the bath adjacent the tank wall and thus eliminates or minimizes risk of sulphate or "rock salt" deposits on the tank wall adjacent the bath level. The addition of air or gas to the steam avoids water hammer effects. No claim is made herein on the arrangement or use of the steam-gas supply pipes MA as such pipes and their use are disclosed and claimed in my prior application Serial No. 475,371 filed February 10, 1943. It is to be noted however, that the pipes MA tend to maintain a bath liquor circulation about the vertical tank axis in the same direction as do the previously mentioned pipes L, and thus contribute to the rapid removal of tar through the overflow outlet nozzle A^6.

Make-up acid is supplied to the saturator through a supply pipe N' and a spray or distributing pipe ring N surrounding the upper inlet portion of the mouthpiece D. Except in respect to the form and disposition of the means for passing the incoming gas into scrubbing contact with the bath liquor, the specific features of the saturator construction and arrangement hereinbefore referred form no part of the invention claimed herein.

As shown in Figs. 3 and 4, the mouthpiece D which may be formed of cast lead, comprises an upper cylindrical portion D', a lower conical lip D^3, and an intermediate portion D^2 outwardly curved or flared to increase in diameter at a progressingly increasing rate as the distance from the lower end of the cylindrical portion D' increases. As shown, the upper end portion of the mouthpiece D' includes a flange D^4 for bolt connection of the mouthpiece to the lower end of the cracker pipe C.

As shown, curved ribs D^5 depending from the underside of the mouthpiece D and extend inward from the periphery of the latter. The ribs D^5 while not essential elements of the present invention, contribute to the proper distribution of the gas passing out of the gas space beneath the mouthpiece and bubbling up through the bath liquor at the mouthpiece periphery. The ribs D^5 also provide curved flow channels for the gas discharged from the cracker pipe and for the bath liquor streams set into motion by the gas and thus supplement the action of the curved discharge ends of the return pipes L and gas injector pipes MA in creating a flow of bath liquor in the counterclockwise direction above the vertical tank axis.

In the normal contemplated use of the apparatus shown in Figs. 1–4, make-up acid is supplied to the saturator through the pipe N as required to maintain the desired bath liquor acidity. The sulphuric acid content of the bath liquor may well be about 5.5 per cent or less in the production of "large" crystals, and about 7 per cent or more in the production of ordinary small size crystals. The actual rate of make-up acid supply depends, of course, upon the rate at which the ammonia containing gas is passed through the saturator and upon the ammonia content of the gas. The level regulating weir K may be adjusted to maintain the top service level B' of the bath liquor at the distance *a* above the lower peripheral corner edge D^6 of mouthpiece D which gives the best operation results under particular operating conditions.

The fact that the use of the present invention increases the operating capacity of saturators has been demonstrated. Thus, for example, a satu-

rator of the type, form and proportions illustrated in Figs. 1–4, in which the diameter of the peripheral edge of the mouthpiece is six and a half feet, and in which the distance *a* is six inches, is now being regularly and successfully used to recover the free ammonia in coke oven gas passing through the saturator at the rate of about forty-four million cubic feet per twenty-four hour day. No prior saturator, as far as I am aware, has ever operated regularly at a higher rate, or at as high a rate per unit of saturator volume or bulk. Said saturator as originally constructed, and as regularly used for a period of years, included means for discharging gas into the bath liquor through vertically elongated ports or slots in vertical walls extending down into the bath liquor far enough so that the upper end of said ports or slots were at a level some fifteen or more inches below the top surface of the bath. While the aggregate horizontal width of said vertical wall slots originally included in the saturator was considerably greater than the circumference of the mouthpiece D, the saturator in its original form was never practically operative at a rate of more than about twenty-two million cubic feet of gas per twenty-four hour day, and its regular use was eventually abandoned because its capacity was too small in comparison with the requirements of the plant in which it was installed and also because its scrubbing performance was not good enough to recover a sufficiently large portion of the ammonia in the gas entering the saturator. With the saturator in its present form and operating at the high rate above mentioned, the ammonia recovery is practically complete. Owing to the much greater distances below the top surface level of the bath at which the gas was discharged into the bath liquor in the original form of the saturator, the gas pumping energy required to move the gas through the saturator was more than twice as great per cubic foot of gas moved as is required in the use of the saturator in its present condition.

In the particular saturator shown in Figs. 1–4, the under-surface of the conical lip D^3 of the mouthpiece D is inclined at an angle of thirteen degrees to the horizontal. With such inclination the maximum gas velocity in the gas space between the mouthpiece and the depressed bath liquor surface B^2 is attained directly beneath the corner edge D^6. The normal or average depth *b* of the portion of the gas space directly beneath the corner edge D^6 depends upon the density of the gas, the density of the bath liquor, the length of the circumferentially extending corner edge D^6 and the quantity rate of gas flow, and can be readily approximated by computation when those factors are known. In the above mentioned saturator operation at the rate of forty-four million cubic feet of gas per twenty-four hour day, the bath liquor density is about 1.3 that of water and the gas density is about 0.389 that of air, the gas space distance *b* is about two and three-tenths inches, and the average linear gas velocity directly beneath the corner edge D^6 is about one hundred twenty-four feet per second.

In normal operation, the gas space depth *b* and the linear gas velocity directly beneath any one particular point along the length of the corner edge D^6 will vary as a result of bath liquor surging and wave movements. An annular outer portion of the surface B^2 normally comprises wave crests B^3 and alternating wave troughs as indicated in the right-hand portion of Fig. 4. The wave crests B^3 are the result of the wave or ripple creating action of the gas moving through the gas space and the height of the wave crests B^3 increases as the periphery of the surface B^2 is approached. As is well known, only a gentle breeze, i. e., a wind velocity of a few miles an hour, is required to maintain the surface of a small pond in a rippled condition. With operation at the rate mentioned above, the minimum gas velocity beneath the lip of the mouthpiece portion D^3 is in excess of a mile a minute. Such a gas velocity is not only sufficient to maintain the subjacent portion of the surface B^2 in a strongly rippled condition, but is sufficient to subject the bath liquor to a substantial atomizing effect. Minute atomized acid particles or droplets swept off the ripply surface B^3 by the high velocity gas steam will be dispersed into the gas stream in a manner insuring highly efficient inter-action between the ammonia constituent of the gas and the acid constituent of the atomized bath liquor.

As a result of studies and tests made by me, I believe that the undersurface of the mouthpiece lip D^3 or analogous gas space roof or top wall should incline downwardly toward the discharge end of the gas space generally as does the lip D^3. As stated above, the inclination to the horizontal of the undersurface of the lip portion D^3 shown in the drawings is thirteen degrees. I found by my practical tests that this angle can be increased to fifteen degrees and can be decreased to four degrees without any radical effect on the general operation of the saturator. I found that above 15° the rippling and atomizing action necessary for the removal of the ammonia began to disappear. I found further by my tests that at an angle smaller than four degrees the bath liquor began to surge. This produces a pulsating gas outflow. Such flow pulsation reduces the efficiency of the scrubbing action and is conducive to objectionable salt deposits on the underside of the mouthpiece lip.

While the exact form and curvature of the intermediate portion D^2 of the mouthpiece is not essential, I do consider it practically important that the curvature of the inner surface of the portion D^2 should be such as to form a rounded orifice inlet along which the gas will flow smoothly instead of being displaced therefrom as a result of a vena contracta action. Such displacement increases the tendency to the formation of objectionable salt deposits on the mouthpiece.

The chart or diagram shown in Fig. 8 illustrates the results of experimental tests made by me to determine effects produced by variations in the velocity of air flow through an air space which in vertical section, is shaped like the gas space beneath the left half of the mouthpiece D shown in Fig. 3. The roof of the said gas space of the apparatus used in making said tests was equal in length or extent measured in the direction of air flow, to the radial extent of the lip portion of a mouthpiece shaped like the mouthpiece D shown in Fig. 3, and having internal and external diameters of three feet and six and a half feet, respectively. In said experimental tests the inclined portion of the gas space roof, corresponding to the conical lip portion D^3 of Fig. 3, was inclined at different angles. In Fig. 8, the abscissae indicate gas space roof wall inclinations in degrees, and the ordinates represent quantitative rates of air flow in cubic feet per minute through a section of the air space beneath the roof wall of the test apparatus having a horizontal width

of one inch measured in a direction transverse to the direction of air flow.

In the tests illustrated in Fig. 8, a noticeable surging action was produced with, and only with, the relatively large rates of air flow and the relatively small inclination to the horizontal of the air space roof wall indicated by points in the chart area at the left of the line 8 of Fig. 8. Relatively deep or strong ripples were created on the water surface beneath the air spaces in the test apparatus with quantity rates of air flow and air space roof inclinations indicated by pc s within the area at the left of the line 9 of Fig. 8. Relatively small but definitely noticeable ripples were created and maintained at the surface of the water beneath the air space in the test apparatus with quantity rates of air flow and air space top wall inclinations indicated by points within the chart area between the lines 9 and 10 of Fig. 8. With quantity rates of gas flow and air space roof inclinations indicated by points in the area at the right of the line 10 of Fig. 8, no significant definite ripple actions were apparent.

In the contemplated operation of the saturator shown in Figs. 1–4, the major portion of the ammonia content of the gas entering the saturator is separated from the gas in its passage through the scrubbing zone gas space under the mouthpiece lip D^3, as a result of the contact of the gas with the turbulent liquor beneath said space, and as a result of dispersion in the gas stream of atomized liquor particles swept off the subjacent liquor and entrained by the gas. The ammonia still held in the gas passing away from the gas space beneath the mouthpiece is eliminated from the gas as the latter bubbles up through the bath liquor in which the mouthpiece lip is submerged.

The effectiveness of the bubbling contact of the gas with the bath liquor may be augmented, and other advantages obtained by providing the mouthpiece lip adjacent its periphery with perforations such as the narrow radially extending slots D^7 shown in Fig. 5. The upflow of gas through the perforations D^7 increases the area of contact of the liquor with the gas bubbling through it and thus contributes to the efficiency of the total scrubbing action. The flow of gas from the gas space beneath the mouthpiece through the perforations D^7 reducing the volume and velocity of the gas passing into the bath liquor at the periphery of the mouthpiece and thus reduces the tendency of the gas to pass into and through the liquor in the form of overly large globules or masses. Such large globules increase the tendency to objectionable surging and impairs the gas and liquor contact efficiency. Furthermore, the reduction in the volume of gas flow into the bath liquor at the periphery of the mouthpiece reduces the amount of entrained bath liquor carried out of the saturator by the gas into the acid catcher. The gas liquor so entrained includes sulphate crystals and subjects the acid catcher to an erosive action desirably avoided.

When the perforations D^7 are adjacent the mouthpiece periphery, as shown in Fig. 5, they do not significantly modify the scrubbing conditions in the gas space beneath the mouthpiece. Moreover, the provision of such perforations may well be accompanied by some increase in the diameter of the mouthpiece. In some cases, a mouthpiece may be provided with perforations serving the purpose of the slots D^7 by attaching an annular perforated peripheral extension to an imperforate mouthpiece body. The distributed discharge of the make-up acid by the pipe N into the liquor above the mouthpiece lip, contributes to the effectiveness of the scrubbing action occurring as the gas bubbles up through the bath liquor, and minimizes any tendency of the perforations D^7 to fill up with deposited sulphate.

In Fig. 6 I have illustrated diagrammatically the general form and proportions of a saturator of a compact and desirable design constructed and operated in accordance with the present invention, and having an operative capacity in excess of ninety million cubic feet of gas per twenty-four day, which is more than double the capacity of any saturator heretofore in use, of which I have knowledge. In the saturator design illustrated in Fig. 6, the saturator tank body A is circular in horizontal cross section and its inside diameter is twenty feet; the inside cracker pipe diameter is nine feet; and the maximum diameter of the cracker pipe mouthpiece DB is thirteen and a half feet. While I believe that generally satisfactory operation of the saturator shown in Fig. 6 will be obtainable with the angle of inclination of the underside of the conical lip of the mouthpiece DB, as great as fifteen degrees or as small as four degrees, I believe that the optimum angle will be about eight degrees if the rate of operation does not vary much from the normal capacity rate of ninety million cubic feet per twenty-four hour day.

In Fig. 7, I have illustrated diagrammatically the use of a vertically disposed tubular element D which is coaxial with and beneath the mouthpiece and surrounds a rotating pump impeller P, such as has been frequently employed in saturators producing "large" sulphate crystals to create a bath liquor circulation to facilitate and augment the growth of sulphate crystals. The element O shown in Fig. 7 differs from the tubular elements heretofore employed in association with an impeller like the impeller P to augment and direct bath circulation, in that its internal diameter progressively increases as its upper end is approached. The diameter of the upper edges or rim portion of the member O is large enough and is located at a suitable distance below the mouthpiece, so that it serves as a baffle, checking and materially minimizing the bath liquor surging tendency.

Saturators constructed and operated in accordance with the present invention may take forms quite different from those already described. Thus, for example, the saturator shown in Figs. 9 and 10 comprises a tank A shaped like that shown in Figs. 1 and 2 but having a cracker pipe element DD in the form of an elongated, inverted trough. The element DD extends horizontally in the direction of the major axis of the elliptical tank A away from the tank wall at one end of said axis, and terminates short of the tank wall adjacent the opposite end of said axis. The crown or upper side of the element is shown as well above the general level of B' of the bath liquor, but the rim portion DD' of the element is submerged. In vertical section transverse to its lower outer edge D^{60}, the rim portion DD' may be proportioned and inclined to the horizontal generally as is the conical lip portion D^3 of the mouthpiece D of Figs. 1 and 3. As shown, the cracker pipe DD receives gas at one end through a suitable inlet in the vertical wall of the tank A from an external gas supply pipe ED, and the inclined rim portion DD' extends circularly about the opposite end of the cracker pipe. At the side of the tank remote from the gas supply connection ED the tank shown in Fig. 9 is provided

with an overflow outlet connection K associated with a tar separator J, as in the construction shown in Figs. 2 and 2a.

The tank saturator shown in Figs. 11 and 12 has a tank structure comprising a lower part AE circular in horizontal cross-section and having a hopper bottom, and comprising an upper circular part AF smaller in diameter and extending down into the upper portion of the part AE and coaxial with the latter. The lower portion of the tank part AF and the upper portion of the tank part AE form the opposite side walls of a gas inlet space CE, which as shown, extends about the vertical axis of the saturator for considerably more than 180°. The space CE is closed at its ends by vertical end walls C^{10}, and is closed at its upper side or edge by a top wall C^{11}. The gas treated is passed into the inlet space CE through a gas supply pipe EE, and the gas passing through the saturator leaves the latter through a discharge pipe FE.

A generally horizontal wall DE extends inwardly from the lower edge of the tank portion AF at the inner side of the gas inlet space CE and forms the roof of a scrubbing zone gas space. The scrubbing zone gas space extends about the vertical axis of the saturator alongside the gas inlet space CE and is in open communication at its outer edge with the space E, and receives gas from the space CE along the full length of the latter. The saturator shown in Figs. 11 and 12 is provided with an overflow outlet K discharging into a tar separator J which may be of the type and form shown in Fig. 2A. Liquor return pipes and steam-gas injection pipes, like the pipes L and MA shown in Figs. 1 and 2, may be employed to create a bath liquor circulation in each of the tanks shown in Fig. 9 and in Figs. 11 and 12. In the operation of any of the saturators shown it is practically desirable to continuously and rapidly remove the tar separating from the gas, since any considerable accumulation at the top of the bath interferes with the scrubbing action and tends to discolor the sulphate crystals formed. The use of means such as those formed by the pipes L and MA for effecting a circulation of bath liquor adjacent to the top of the bath tends to expedite the removal of the tar as well as to augment the efficiency of the sulphate forming process. The regulation of the bath level effected by the vertical adjustment of the weir K permits of a desirable regulation of the character of the circulation produced by the jets discharged through the pipes L and MA.

I have discovered that when the ammonia containing gas is discharged into the bath liquor at the relatively high quantity rate made readily possible by the hereinbefore described method of passing gas into scrubbing contact with the bath liquor, the tendency to the deposit of sulphate crystals on the wall of the gas space above the bath liquor is substantially reduced. In consequence, the amount of wash liquid used in the general manner described in my prior application Serial No. 473,949, filed January 29, 1943, to prevent such deposits, may be substantially smaller than when the rate at which gas is discharged into the bath liquor is at the lower rates. Thus, for example, heretofore customarily in the operation of the above mentioned saturator constructed as shown in Figs. 1–4, I have found that when gas is passed into the saturator at the rate of about forty-four million cubic feet of gas per twenty-four hour day, objectionable deposits of crystallized sulphate on the gas space wall may be substantially avoided without using more than a relatively small fraction of the wash liquor needed to prevent such deposits when the same saturator is operated at the rate of twenty-two million cubic feet of gas per twenty-four hour day.

I believe that the operation of the saturator at the high rate mentioned minimizes or prevents sulphate crystal deposits on the gas space wall as a result of the relatively large amount of bath liquor splashed onto the gas space walls by the geyser-like stream of gas bubbling up through the bath liquor from the discharge edge of the scrubbing zone gas space. That gas flow is operative to maintain a body or mass B^4 of bath liquor and gas above the discharge edge D^{16} of the cracker pipe mouthpiece DB which extends above the top level B' of the unemulsified bath liquor generally as illustrated at the right-hand side of the mouthpiece DB shown in Fig. 6. The gas moving rapidly through and out of the emulsified liquor body B^4, splashes liquor against the gas space wall at a rate appreciably in excess of the rate at which bath liquor adhering to the wall is evaporated by the gas flowing through the gas space. In consequence, a down moving film of bath liquor is maintained on the gas space wall which carries with it its sulphate crystal content of said film.

My observations of the operation of the above mentioned saturator shown in Figs. 1–4, leads me to believe that the optimum practical advantage obtainable by splashing bath liquor onto the gas space wall in a saturator of the general types and forms illustrated, will ordinarily be obtained when the quantity rate of gas flow out of the scrubbing zone gas space and into the bath liquor, is in excess of two cubic feet, preferably about two and an eighth cubic feet, per second, per inch of length of the discharge edge of the roof above said gas space, i. e., the edge designated D^6 in Fig. 4, D^{16} in Fig. 6, D^{60} in Figs. 9 and 10, and D^{66} in Fig. 12.

When the body B^4 of bath liquor above the discharge edge of the scrubbing zone space is annular, as is the case when the cracker pipe mouthpiece of the forms shown in Figs. 4 and 6, for example, it tends to the maintenance of a top surface level of the non-emulsified bath liquor which is slightly higher at the outer side of the body B^4 than at the inner side of said body. This variation in liquor surface levels at opposite sides of the body B^4 of emulsified bath liquor above the discharge edge of the scrubbing zone gas space, can be substantially eliminated by suitably interrupting the continuity of the emulsified bath liquor body. Such interruption is obtained, for example with the construction shown in Figs. 11 and 12, in which bath liquor has an opportunity to flow into the space above the arc-shaped wall DE from the space between the ends DE of that wall. As will be readily apparent, the continuity of the body B^4 shown in Fig. 7 could be similarly interrupted by thickening the outer end portions of some of the ribs D^5, particularly if said thickened ribs be extended outward beyond the periphery of the mouthpiece lip as shown in Fig. 7.

While in accordance with the provisions of the statutes, I have illustrated and described the best forms of embodiment of my invention now known to me, it will be apparent to those skilled in the art that changes may be made in the forms of the apparatus disclosed without departing from the spirit of my invention as set

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forth in the appended claims, and that in some cases certain features of my invention may be used to advantage without a corresponding use of other features.

Having now described my invention, what I claim as new and desire to secure by Letters Patent, is:

1. A saturator for producing ammonium sulphate comprising in combination an enclosed tank, an overflow outlet in the wall of said tank for establishing and maintaining the level of liquor therein, a crackerpipe having a lower vertical portion centrally disposed in said tank and having an outwardly downwardly flared mouth at its lower end, the rim of said flare mouth being at a level slightly below the overflow level and the flared portion having a downward slope from the crackerpipe toward the rim of from 4° to 15° to the horizontal.

2. A saturator as claimed in claim 1 with the addition that slots are provided near the rim in the flared mouth of the crackerpipe.

3. In a process for producing ammonium sulfate which comprises passing ammonia-containing gas into contact with a bath liquor containing sulfuric acid, the improvement which consists in passing the gas beneath a surface which

slopes downwardly, from the point at which it is brought in, at an angle of 4° to 15° to the horizontal, the lower edge of said surface being below the normal liquid level, so that the liquid surface beneath the sloping surface is depressed and agitated by said gas, the rate of gas flow being in excess of two cubic feet per second per inch of length of the discharge edge of said surface.

CARL OTTO.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,986,900	Sperr	Jan. 8, 1935
2,123,887	French	July 19, 1938
1,012,273	Pennock et al.	Dec. 19, 1911

FOREIGN PATENTS

Number	Country	Date
361,935	Breat Britain	Nov. 23, 1931
648,540	Germany	Aug. 3, 1937
272,601	Germany	Apr. 4, 1914
657,439	Germany	Mar. 4, 1938

Plaintiff's Exhibit 5.

October 20, 1947.

Harold C. Cox
R. E. Hough, General Manager
Otto Saturator for Franklin Coke Plant

In a letter to you July 28, 1947, concerning the above subject the writer recommended the purchase of an Otto saturator similar to the ones now in operation at the Bethlehem Coke Plant.

About the same time this recommendation was made the Lackawanna Plant put into operation a new spray type Otto saturator, which is different from the Bethlehem Plant cracker pipe Otto saturator. The Lackawanna saturator is the only one of its kind in existence. It was built so that if with the principle of spray scrubbing the gas failed the saturator could be converted, with the installation of a cracker pipe, to the same type as the Bethlehem Plant. The Lackawanna Plant was thus protected in a new process put into operation for the first time.

The writer has seen the Lackawanna saturator in operation and from all evidence obtained the principle of spray scrubbing the gas is a success and it will not be necessary to install a cracker pipe in the Lackawanna saturator.

With the facts now known of the Lackwanna Plant saturator the Otto Construction Company has offered the Johnstown Plant a new proposition, which is attached hereto.

It has been calculated the following savings are possible if the new Otto saturator will be installed at the Franklin Coke Plant:

1) Annual labor savings of	$32,000.00
2) " power " "	44,500.00
Total Savings	$76,500.00

Plaintiff's Exhibit 5.

The price quoted by the Otto Construction Company for the complete installation of the new type saturator is $258,000. The savings of $76,500 per year in labor and power will pay off the $258,000 expenditure in less than 3½ years.

The condition of the present old saturators at the Franklin Coke Plant demands immediate action on this proposition. The condition of the saturators is as follows:

1) No. 1 was relined with lead 13 years ago after 11 years of previous service. Its lining is very bad now—it is leaking badly and will have to have a new cast iron conical bottom and new concrete foundation, along with a complete new lead lining.

2) No. 2 saturator was relined with lead 10 years ago after 14 years of previous operation. Its lead lining is in very poor condition and its heavy lead cracker pipe is very bad at the present time.

3) No. 3 saturator was seldom run until 1934. It has its original lead lining, which is in poor condition because it has operated steadily for the past 13 years. Its cracker pipe was completely worn out and was replaced by one taken from No. 4 saturator.

4) No. 4 saturator was seldom run. It has its original lining but has no cracker pipe since it was moved to No. 3 saturator. All sections of No. 4 leak and the lead work is badly bulged. This unit needs complete relining.

It is estimated it will require approximately $60,000 to repair the four old saturators. These repairs will have to start promptly if the new Otto saturator is not purchased. If the old saturators are repaired there will be no labor savings or power savings as compared to the new type

Plaintiff's Exhibit 5.

Otto saturator. It is believed unwise to perpetuate the old saturators by spending $60,000 to repair them when a new saturator will pay for itself by saving 76,500 in labor and power each year.

It is necessary to proceed promptly with either the new saturator or repairs to the old ones or suffer heavy ammonium sulphate losses when the new No. 19 Wilputte battery of ovens goes into operation one year from now. The present sale price of sulphate is $35.33 per ton. The present cost of manufacturing the sulphate is running approximately $32.29 per ton. There is a profit of $3.04 per ton even under present operating conditions which are expensive due to the poor condition of the equipment. The present saturators will not handle any more gas unless completely overhauled. Therefore when the new No. 19 Wilputte battery of ovens goes into operation a year from now and 15,000,000 cubic feet of gas additional is available for sulphate manufacture a loss of 376 tons of sulphate will occur per month or at $3.04 per ton profit will amount to $1,143.00 per month. This loss will occur unless the present old type saturators are rebuilt or new Otto saturators are purchased and installed before the new battery of ovens is started.

In order to realize savings and avoid losses it is urged the Otto saturator be considered promptly and favorably.

HAROLD C. COX,
Superintendent of Coke Plants.

Plaintiff's Exhibit 8.

LECTURE No. 7—COURSE K-171
COKE OVEN PLANT DESIGN
BY-PRODUCT EQUIPMENT

N. AMMONIUM SULPHATE—GENERAL

1. Amount of sulphate produced per ton coal carbonized, approximately 24 lbs. per ton.
   a. Varies with temperature of carbonization.
      1. Low temperature carbonization does not produce ammonia for conversion to sulphate.
      2. Cool oven tops result in increased sulphate production whereas hot oven tops reduce sulphate yield. Tests by Bureau of Mines indicate optimum sulphate production at cooking temperatures of around 1700° F. (measured in oven).
   b. Depends upon type of coal.
      1. Nitrogen content.
         a. Ammonia produced represents about 10 to 20% of nitrogen in the coal.
      2. Moisture content.
         a. The higher the moisture content the more sulphate that is produced.
2. Processes of Recovery.
   a. Direct.
      1. Now obsolete due primarily to operating difficulties.

Plaintiff's Exhibit 8.

a. Consisted of contacting hot gas with sulphuric acid.

1. Hydrogen chloride—main trouble.

2. Tar recovery disturbed.

b. Semi-direct.

1. Operates satisfactorily in almost all sulphate plants.

2. Comprises contacting ammonia laden gas with sulphuric acid.

a. Ammonia from liquor returned to gas.

3. Chemical reactions and calculations involved:

$2\ NH_3$ (ammonia) + H_2SO_4 (sulphuric acid) = $(NH_4)_2SO_4$ (ammonium sulphate).

or by molecular weights:

34# ammonia + 98# of acid = 132# of sulphate.

For every lb. of ammonia $\frac{132}{34}$ =

3.88# of sulphate is formed.

For 6 lbs. of NH_3 per ton of coal carbonized—6 x 3.88 = 23.3# of sulphate is formed.

c. Indirect.

Plaintiff's Exhibit 8.

1. Used to some extent in Europe.
2. Consists of washing gas with water by means of ammonia scrubbers; adding this liquor to that obtained in the primary cooler system, then distilling and treating the vapors so produced in an indirect saturator.
3. Different materials of construction are required in an indirect saturator since the heat of reaction keeps the bath at boiling temperatures.
4. Steam or coke oven gas is used to agitate mother liquor bath.
5. Care must be taken to condense vent gases or discharge them to a gas main because they contain H_2S, HCN, and organic vapors along with steam, CO_2, etc.

O. SATURATORS.

1. Various Types.
   a. Old cracker pipe type.
      1. Lead lined.
      2. High pressure drop around 30″ H_2O.
      3. High construction cost.
      4. Subject to high maintenance.
   b. Multiple dip pipe type.
      1. Built in World War II days.
      2. High pressure drop around 20″ H_2O.

Plaintiff's Exhibit 8.

3. Construction—lead lined with brick and stainless steel.

4. Propeller for agitation.

c. Bustle pipe type (e. g. Seaboard, Aliquippa).

1. Bustle pipe around periphery within the saturator and slotted for gas entrance into bath.

2. Unbalanced because of swishing of the bath from one level to another.

3. Vibration likely to be excessive, particularly in larger diameters.

4. High pressure drop around 20″ H_2O.

d. Center dip pipe type—Otto (e. g. Midland, Republic-Warren).

1. Medium pressure drop around 12 to 15″ H_2O.

2. Low cost for size.

3. High ratings possible.

4. Makes very fine crystals.

e. Spray type.

1. Neville Island, Woodward, Sloss Sheffield, Kaiser, etc.

2. Advantages of Spray Type.

a. Low pressure drop 3″ to 6″ H_2O pressure.

b. High removal of NH_3 (ammonia) 99.7% or better.

c. Good salt size—can be controlled.

Plaintiff's Exhibit 8.

d. Can be built with or without combined acid separator.

e. Less ground space required (smaller diameter for equivalent production) 18′ spray type equivalent to 25′ bustle pipe type.

f. Less vibration—no surging of bath since gas does not bubble through bath.

g. Uniform crystal size.

3. Functions of Saturator.

a. Remove ammonia from gas.

b. Produce ammonium sulphate.

c. Possible removal means for pyridine.

d. Classification of sulphate crystals for maximum size.

e. Reservoir for mother liquor and slurry.

4. Design of Spray Type Saturator.

a. Capacity.

1. 12′-0″ diameter good for 30,000,000 C. F. D. gas.

b. Height about 34′-0″.

c. Combined acid separator.

d. Classification bowl.

e. Spray chamber design.

1. All portions wetted.

2. Importance of Sprays.

f. Stainless steel should contain less than 06 carbon.

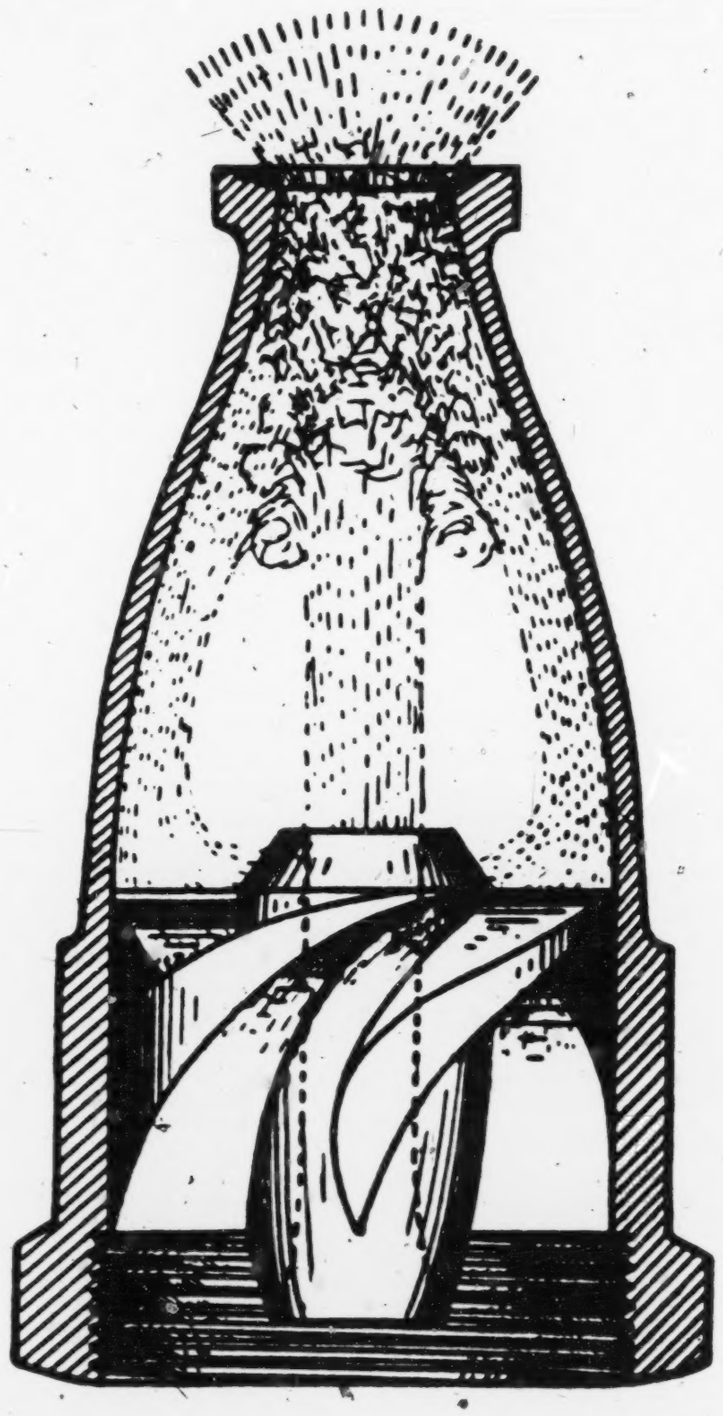
Spraco Center-jet Nozzles

A great many spray applications require a nozzle which will deliver a full cone spray, that is a spray which provides a uniform distribution over the entire area covered.

Spraco Engineers developed the center-jet principle and applied it to a nozzle design in order to produce this full cone or solid type of spray.

The sectional view shows the action which takes place within the nozzle. The major portion of the liquid passes through the outer vanes of the stationary turbine center and is given a rapid swirling motion.

The remainder of the liquid passes straight through the center-jet and strikes the outer rotating mass just below the orifice in the mixing chamber, causing the liquid to issue from the nozzle in a full conical formation of equal density across its section.



This patented type of nozzle is particularly recommended for processes where an absolutely uniform spray is required. The distribution of drops is so even that every portion of the entire area covered receives the same amount of the liquid sprayed.

The size of the drops will depend upon the size of the nozzle and the pressure under which the liquid is sprayed. The full cone effect is produced even at relatively low pressures making this nozzle particularly well adapted to spray cooling systems and aeration work.

In the construction of Spraco center-jet nozzles the turbine center is a separate part, allowing the generation of a vane design which insures maximum operating efficiency.

The removable turbine center allows the characteristics of the spray to be changed without furnishing a complete new nozzle.

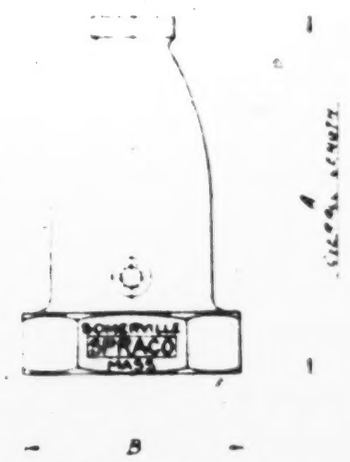
The accompanying table furnishes complete data on the center-jet full cone Spraco nozzles, including capacities and spray characteristics under different conditions of water pressure up to 100 lbs.

Materials used in the manufacture of stock nozzles include brass, bronze, steel and cast iron. Nozzles can be furnished made from a wide variety of other materials including monel metal, stainless steel, hard rubber, bakelite, antimonial lead and special alloys.

In addition to the center-jet nozzles listed below the Spraco line includes three turbine center type nozzles designated as 1-A, 1-S and 6-H made with a turbine center but not having the center-jet, these nozzles producing a hollow cone spray. Capacities and spray characteristics for these three nozzles are listed in the supplementary table at the bottom of this page.

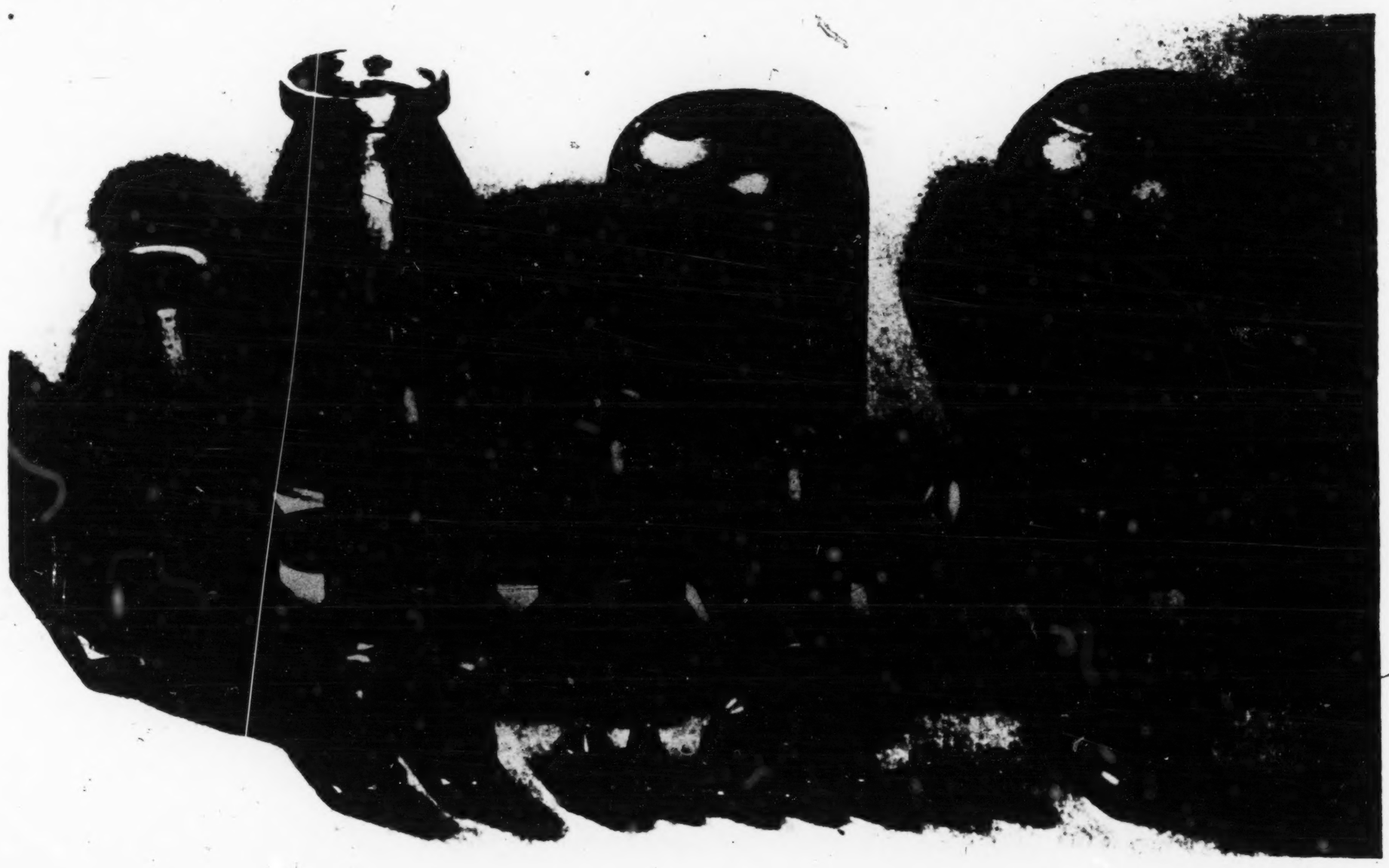
PARTIAL LIST OF USES

Aerating and purifying water supplies
Cooling circulating water for condensers
Spraying brine for refrigeration purposes
Quenching coke and pig iron
Washing coal, sand and gravel
Beating down foam in paper manufacture
Atomizing various liquids in chemical manufacture
Distributing oil over the fuel bed in gas machines
Manufacturing salt by spray evaporation
Protecting inflammable processes from fire



Size No.	Pipe Thread Connection Inches		Dia. of Orifice	Large Dia. Bronze	Overall Length Inches Bronze		Net Wt.	Pressure 5 Lbs. Per Square Inch			Pressure 10 Lbs. Per Square Inch			Pressure 15 Lbs. Per Square Inch			Pressure 20 Lbs Per Square Inch		
	Male	Fem.	Inches	Inches	Male	Fem.	Oz.	G.P.M.	Angle	Hght.	G.P.M.	Angle	Hght.	G.P.M.	Angle	Hght.	G.P.M.	Angle	Hght.
1B	1/4	1/4	3/64	7/8	1 3/8	1 3/16	1 1/2										.30	60°	4'0"
2B	1/4	1/4	1/16	7/8	1 15/32	1 11/32	1 3/4										.46	45°	5'0"
3B	1/4	1/4	5/64	7/8	1 5/8	1 1/2	2							.40	45°	4'0"	.46	45°	5'0"
4B	3/8	3/8	.089	1 1/32	1 23/32	1 11/16	2 1/2							.60	50°	4'0"	.69	50°	5'0"
5B	3/8	3/8	3/8	1 1/32	1 23/32	1 11/16	2 1/2							1.18	65°	4'0"	1.36	65°	5'0"
6E	1/2	1/2	3/16	1 3/32	1 25/32	1 21/32	3 1/2	1.15	55°	2'0"	1.60	55°	4'0"	1.94	55°	5'0"	2.22	58°	5'6"
6F	1/2	1/2	3/16	2	1 25/32	1 21/32	3 3/4	1.15	55°	2'0"	1.60	55°	4'0"	1.94	55°	5'0"	2.22	58°	5'6"
6K	1/2	1/2	1/4	2	1 3/4	1 5/8	3 3/4	2.25	48°	3'6"	3.10	53°	6'6"	3.78	55°	7'6"	4.30	60°	8'0"
7B		3/4	1/4	1 13/32		2 25/32	7	3.72	57°	2'6"	5.20	64°	5'0"	6.30	68°	7'0"	7.30	72°	7'0"
8A		1	5/16	1 3/4		2 31/32	9 1/4	5.60	78°	2'6"	7.70	79°	5'0"	9.30	80°	7'0"	10.60	77°	7'6"
9A		1 1/4	3/8	2 5/16		3 3/4	21	8.20	73°	2'6"	11.3	80°	5'0"	13.7	83°	8'0"	15.7	83°	8'6"
10A		1 1/2	1/2	2 17/32		4 1/8	23	12.50	87°	4'6"	17.5	86°	5'6"	21.3	85°	6'6"	24.6	84°	7'0"
10BA		1 1/2	9/16	2 17/32		4 1/4	28	19.5	82°	4'6"	26.0	82°	6'6"	31.0	82°	7'6"	35.0	81°	8'6"
11A		2	25/32	3 3/16		4 7/8	30	33.3	82°	4'6"	47.5	82°	7'6"	58.2	82°	9'0"	67.5	82°	10'0"
11BA		2	1	3 3/16		5 1/8	35	53.0	100°	5'0"	71.5	100°	7'0"	84.5	100°	10'0"	95.0	100°	11'0"
12B		2 1/2	1 1/16	3 3/4		6 3/8	70	75.0	81°	5'6"	99.0	81°	9'6"	116.0	81°	12'6"	130.0	81°	15'6"
13A		3	1 5/16	4 1/2		7 3/4	105	100.0	73°	6'6"	140.0	73°	12'0"	170.0	72°	16'0"	195.0	70°	22'0"
15A		4	1 3/4	5 3/4		9 3/8	203	165.0	82°	5'6"	233.0	82°	9'6"	283.0	80°	15'0"	325.0	80°	19'0"
15D		4	2 9/64	5 3/4		7 3/16		183.	97°		265.	98°		330.	99°		380.	100°	
16A		5	2 5/16	7 1/2		6 3/4		290.	89°		412.	90°		500.	92°		575.	92°	
17A		6	3 13/32	8 11/16		10 1/2		485.	89°		695.	92°		840.	93°		960.	93°	
1A	1/4	1/4	.020	7/8	1 3/8	1 1/4	1 1/2												
1S	1/4	1/4	.025	7/8	1 3/8	1 1/4	1 1/2												
6H	3/8	3/8	17/64	2	1 3/4	1 9/16	3 1/4	.72	80°	18"	1.15	82°	2'6"	1.4	85°	2'6"	1.62	87°	3'0"

p 279 (3)



The above group of Spraco center-jet nozzles graphically illustrates the wide range of sizes carried in stock. The smaller sizes up to and including the No. 6-K nozzles consist of three parts, shell, base and center. This allows replacement of the shell if the orifice becomes worn or distorted. The larger sizes have only two parts, the shell and the turbine center.

Pressure 25 Lbs Per Square Inch			Pressure 30 Lbs Per Square Inch			Pressure 40 Lbs. Per Square Inch			Pressure 50 Lbs. Per Square Inch			Pressure 75 Lbs Per Square Inch			Pressure 100 Lbs. Per Square Inch		
G P M	Angle	Height	G P M	Angle	Height	G P M	Angle	Height	G P M	Angle	Height	G P M	Angle	Height	G P M	Angle	Height
.167	65	2'6"	.18	65°	3'0"	.21	70°	3'6"	.235	70°	5'0"	.29	70°	6'0"	.34	70°	6'6"
.330	65	5'0"	.36	65°	5'6"	.42	70°	6'0"	.470	70°	6'6"	.56	70°	7'0"	.65	70°	7'6"
.510	45	5'6"	.56	48°	6'0"	.65	50°	6'6"	.730	50°	7'0"	.89	50°	7'0"	1.02	50°	7'0"
.765	50°	5'6"	.84	52	5'6"	.95	52°	5'6"	1.06	50°	6'6"	1.30	50°	7'0"	1.49	50°	7'0"
1.50	68°	5'0"	1.65	73°	5'6"	1.89	75°	6'0"	2.08	75°	7'0"	2.50	75°	7'6"	2.88	75°	7'6"
2.46	58°	7'0"	2.68	60°	7'6"	3.08	60°	8'6"	3.42	60°	8'6"	4.15	60°	8'6"	4.78	60°	8'6"
2.46	58°	7'0"	2.68	60°	7'6"	3.08	60°	8'6"	3.42	60°	8'6"	4.15	60°	8'6"	4.78	60°	8'6"
4.80	63°	9'0"	5.25	63°	9'6"	5.00	65°	10'0"	6.65	65°	11'0"	8.10	65°	12'0"	9.30	65°	13'0"
8.15	72°	7'0"	8.80	71°	7'6"	10.25	68°	10'0"	11.30	68°	12'0"	13.70	68°	13'0"			
11.80	73°	9'0"	12.80	70°	10'6"	14.60	68°	13'6"	16.20	66°	15'0"						
17.5	83°	9'6"	19.0	84°	9'0"	21.7	84°	10'0"	24.2	83°	11'0"						
27.50	84°	8'0"	30.0	83°	12'0"	34.5	75°	14'0"	38.70	73°	15'0"						

SPRACO NOZZLES

A wide angle, full cone spray produced by a Spraco Center-jet Nozzle. Note the uniform distribution of spray. Center-jet nozzles can be supplied to produce any intermediate shapes desired between this spray and the one shown just below.

A long, narrow angle. full cone spray produced by a Spraco Center-jet Nozzle. This type of spray is desired for aeration where fine atomization and height give maximum contact with the air.

Spraco nozzles spray equally well in any position. The spray produced by a 15-D Center-jet Nozzle delivering 270 gallons of water per minute at 10 lbs. pressure.

SPRAY ENGINEERING CO. SOMERVILLE, MASS., U. S. A.
BULLETIN No. 602G
3M-6-47 *Printed in U. S. A.*

P. 381 (1)

July 8, 1947. C. OTTO 2,423,794

PRODUCTION OF AMMONIUM SULPHATE

Filed Aug. 25, 1943 3 Sheets-Sheet 1

Fig. 1.

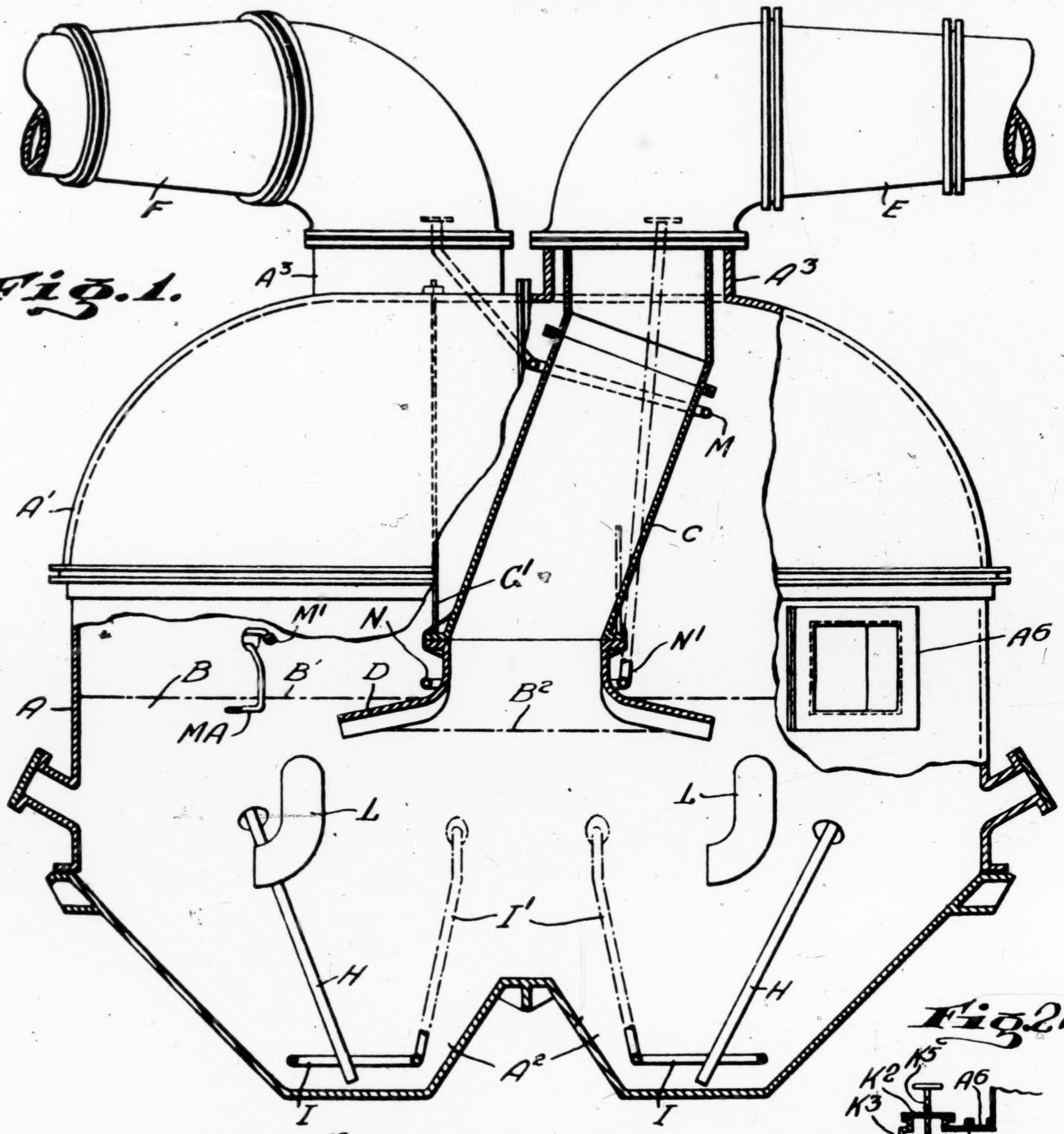


Fig. 2.

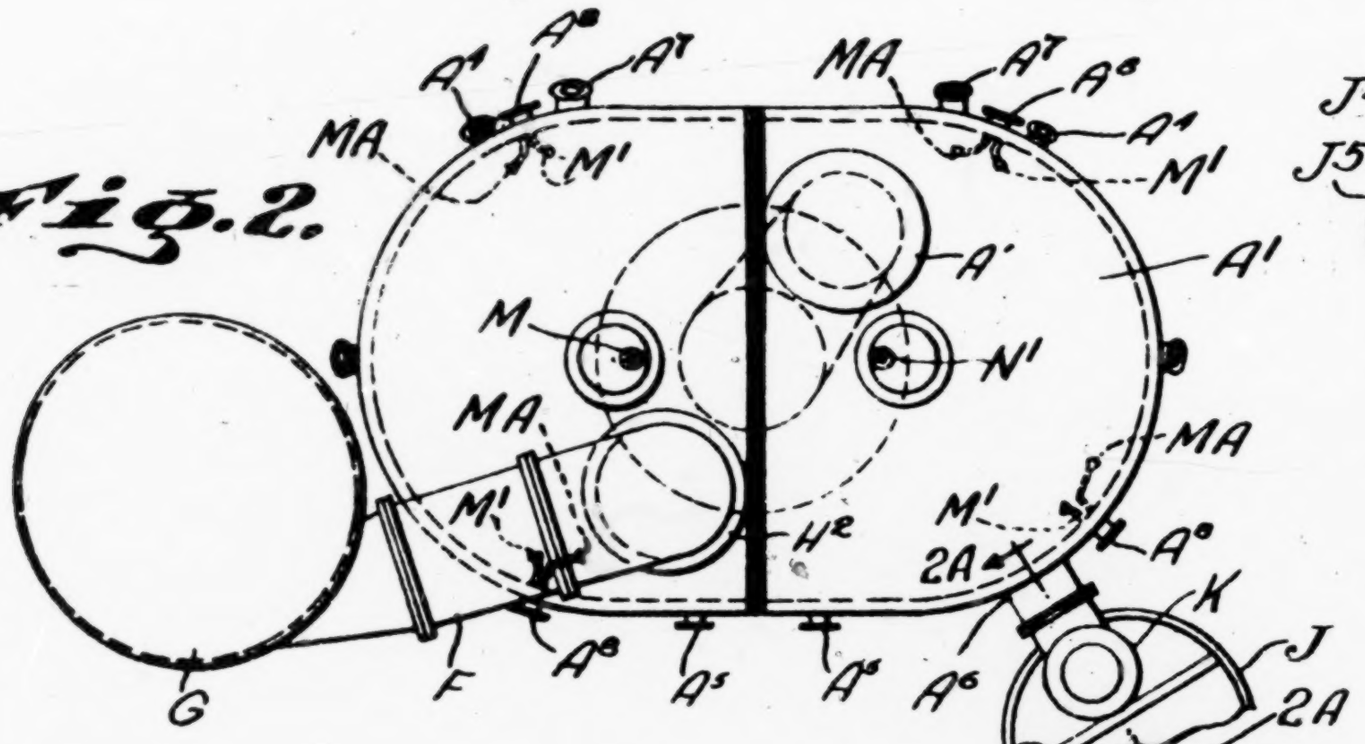
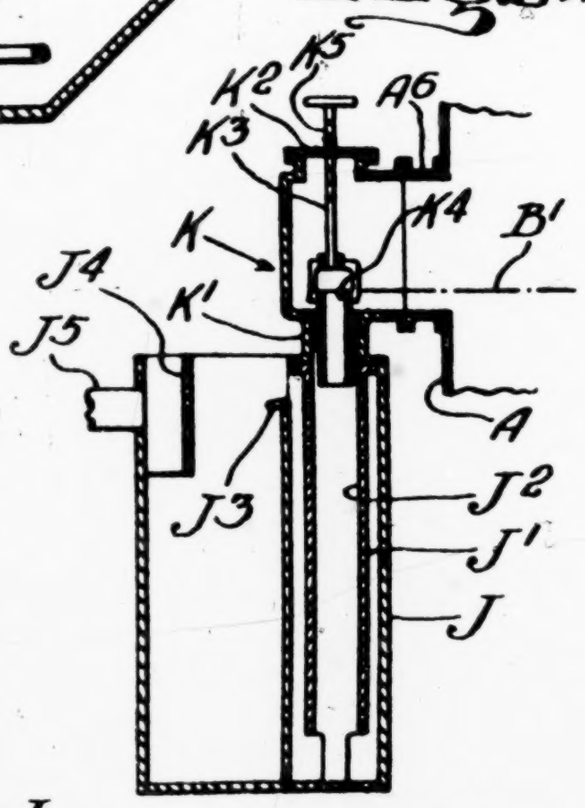


Fig. 2A.



INVENTOR
CARL OTTO

BY John E. Hubbell
ATTORNEY

July 8, 1947. C. OTTO 2,423,794

PRODUCTION OF AMMONIUM SULPHATE

Filed Aug. 25, 1943 3 Sheets-Sheet 2

Fig. 3.

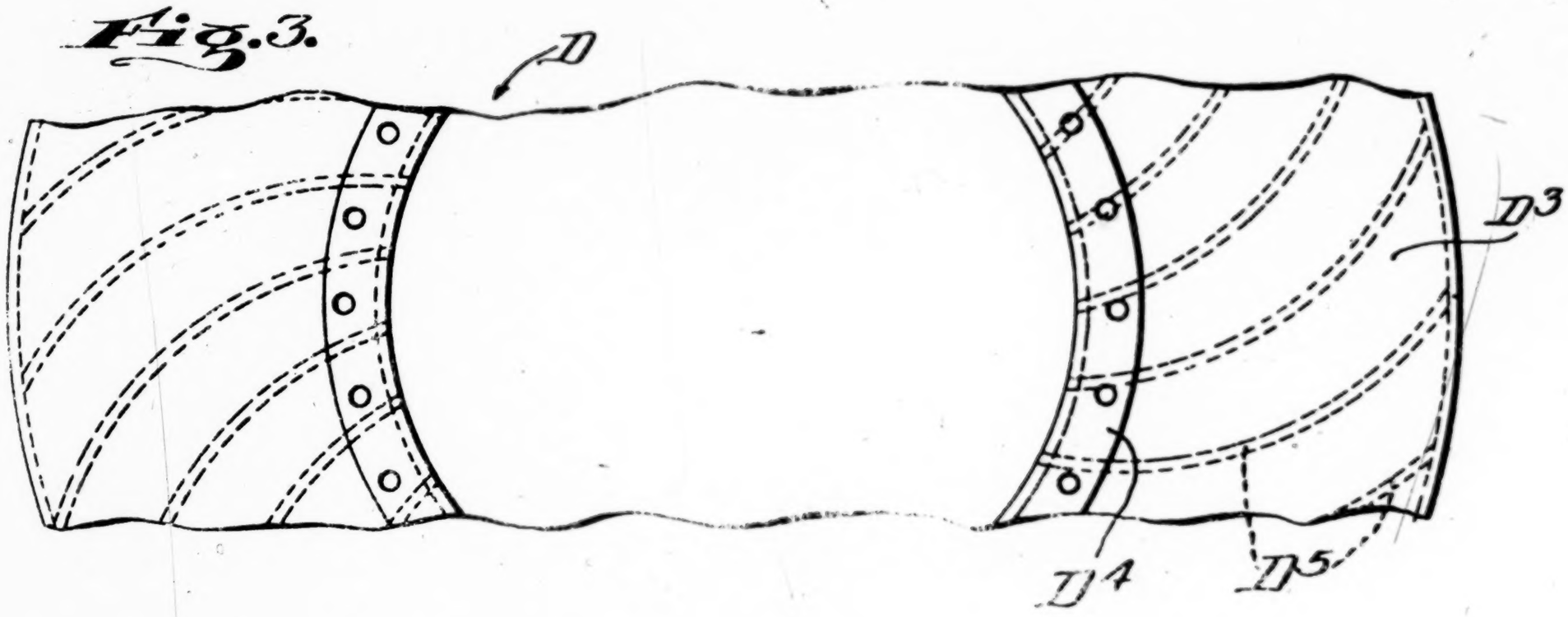


Fig. 4.

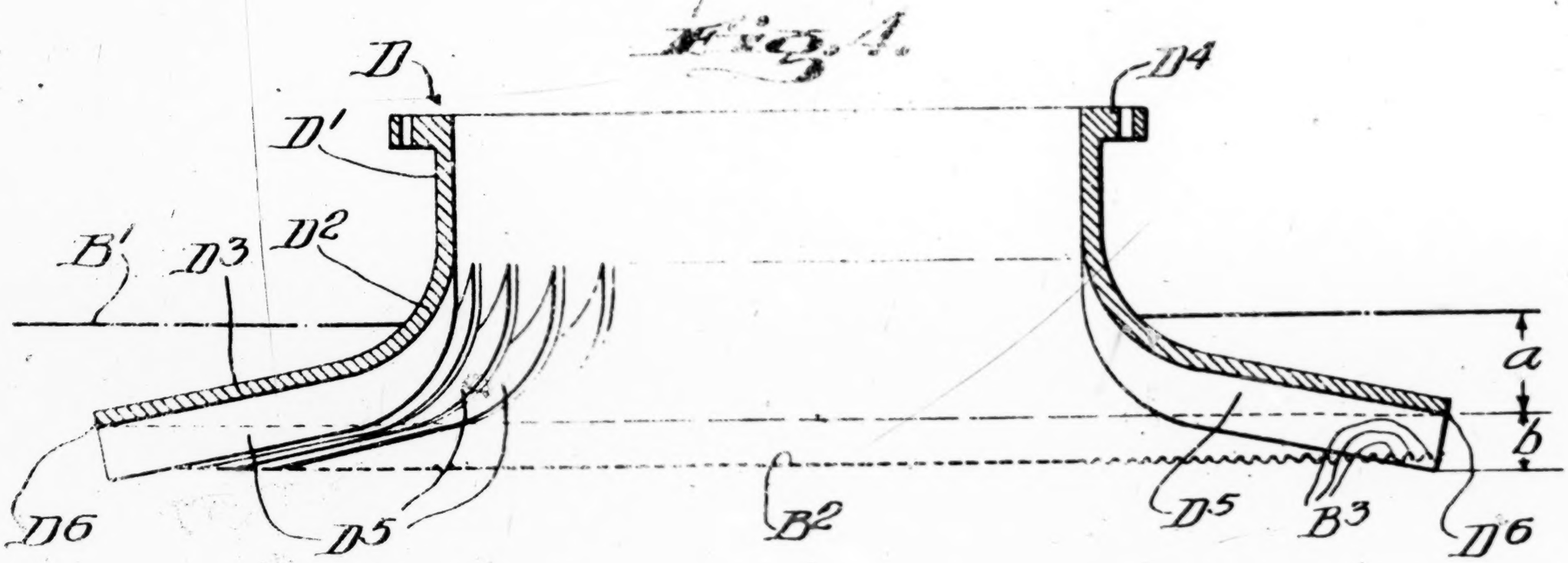


Fig. 5.

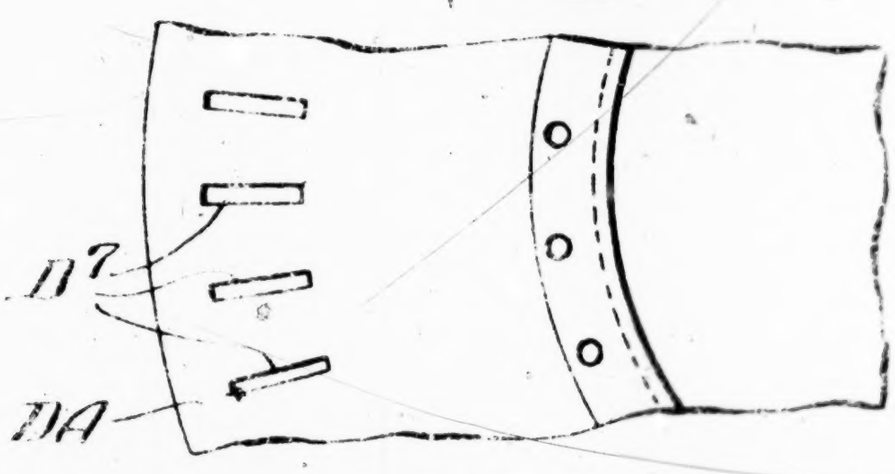


Fig. 7.

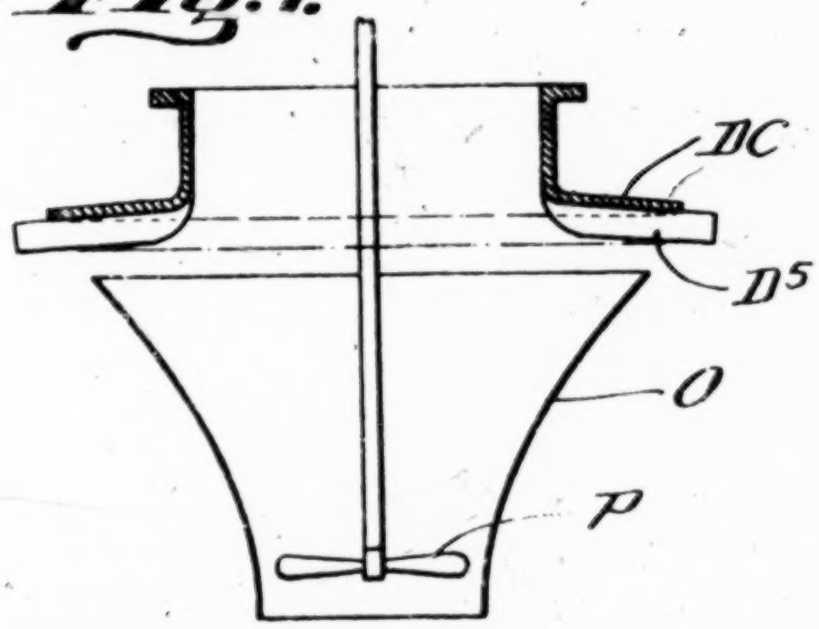
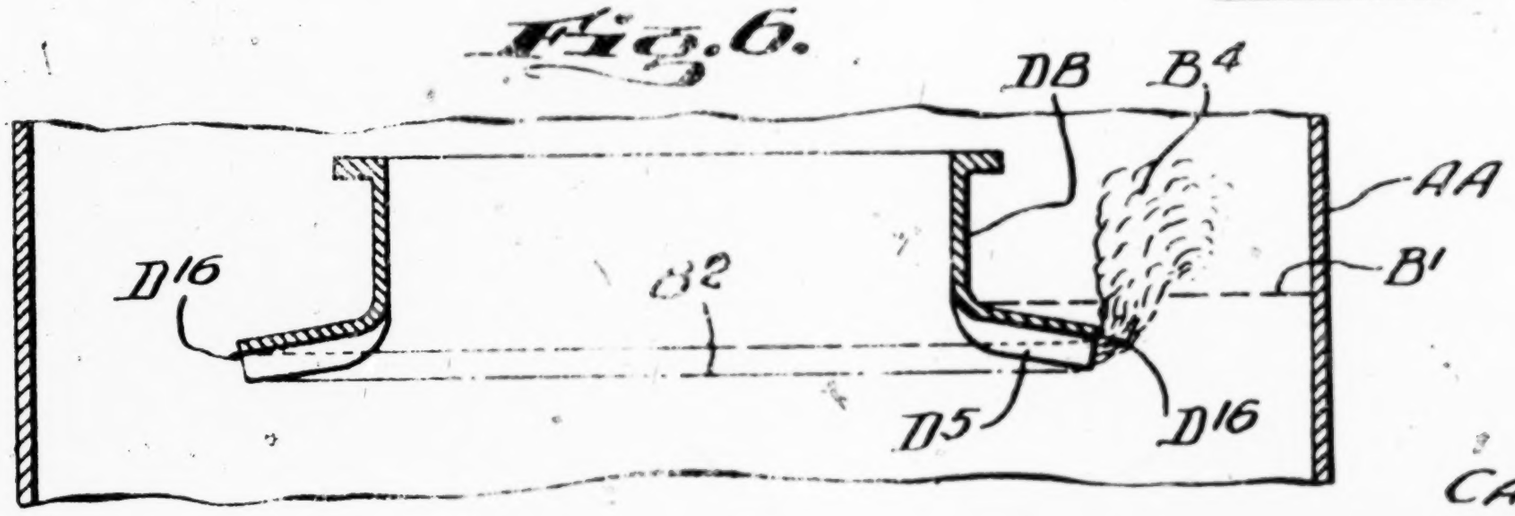


Fig. 6.



INVENTOR
CARL OTTO

BY John E. Hubbell

ATTORNEY

July 8, 1947. C. OTTO p. 381 (3) 2,423,794

PRODUCTION OF AMMONIUM SULPHATE

Filed Aug. 25, 1943 3 Sheets-Sheet 3

Fig. 9.

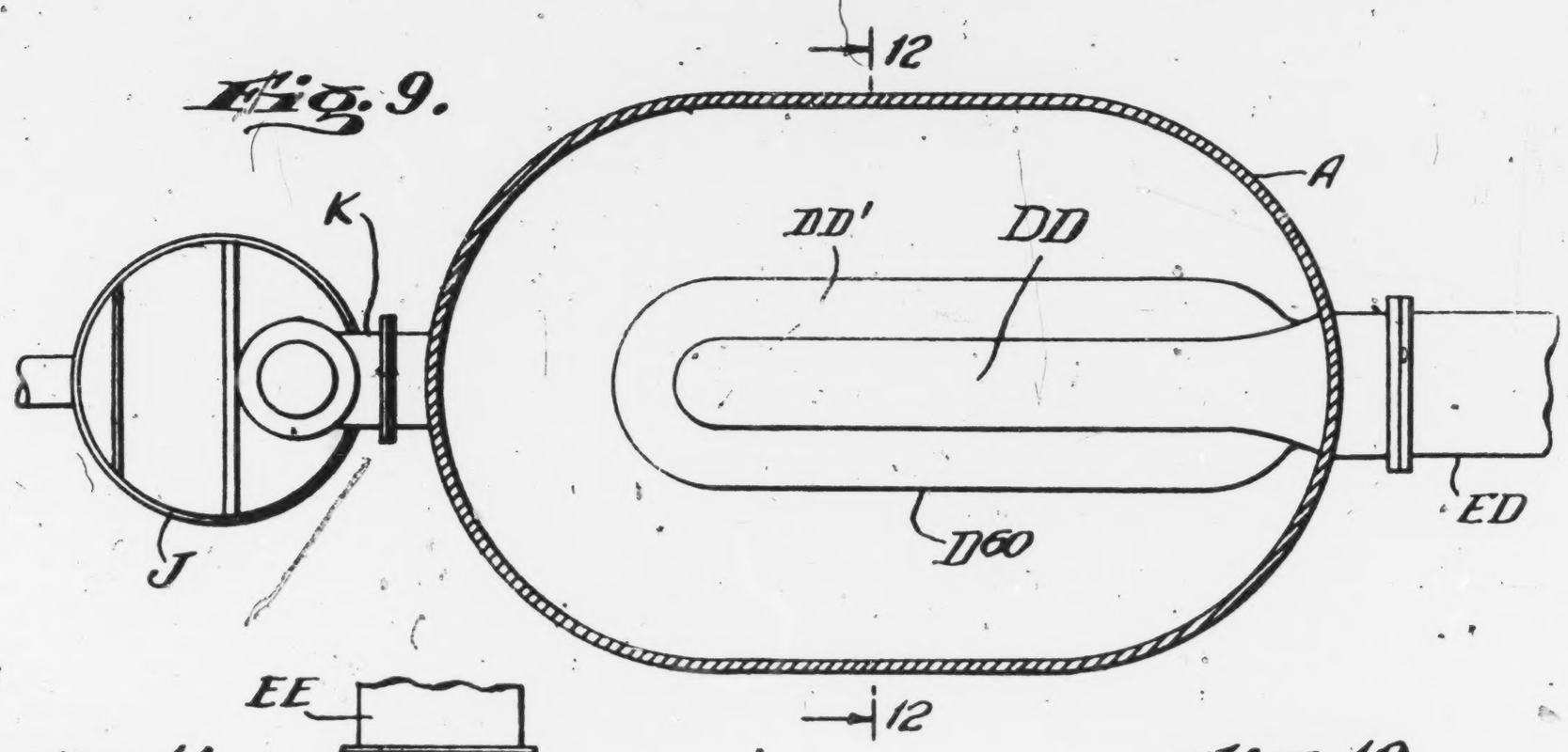


Fig. 11.

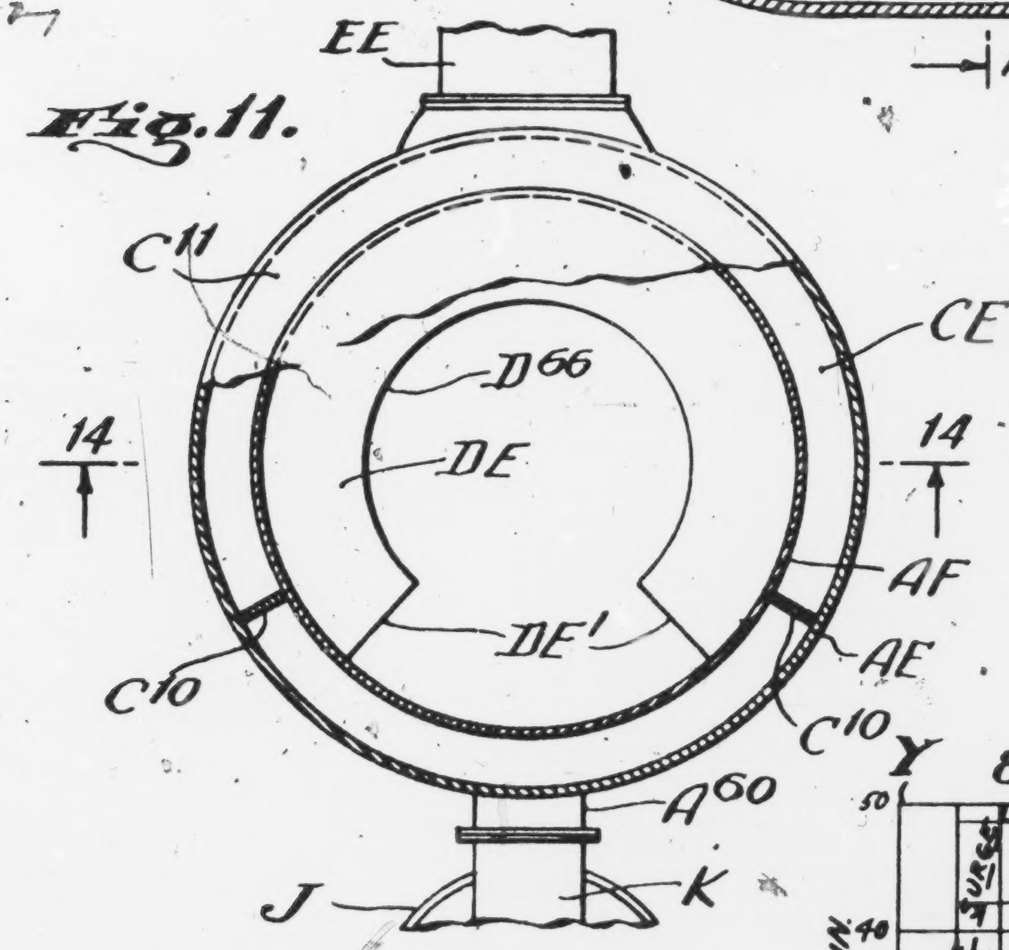
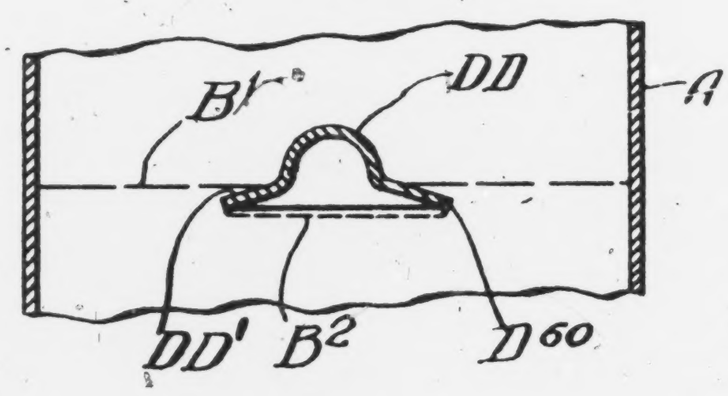


Fig. 10.



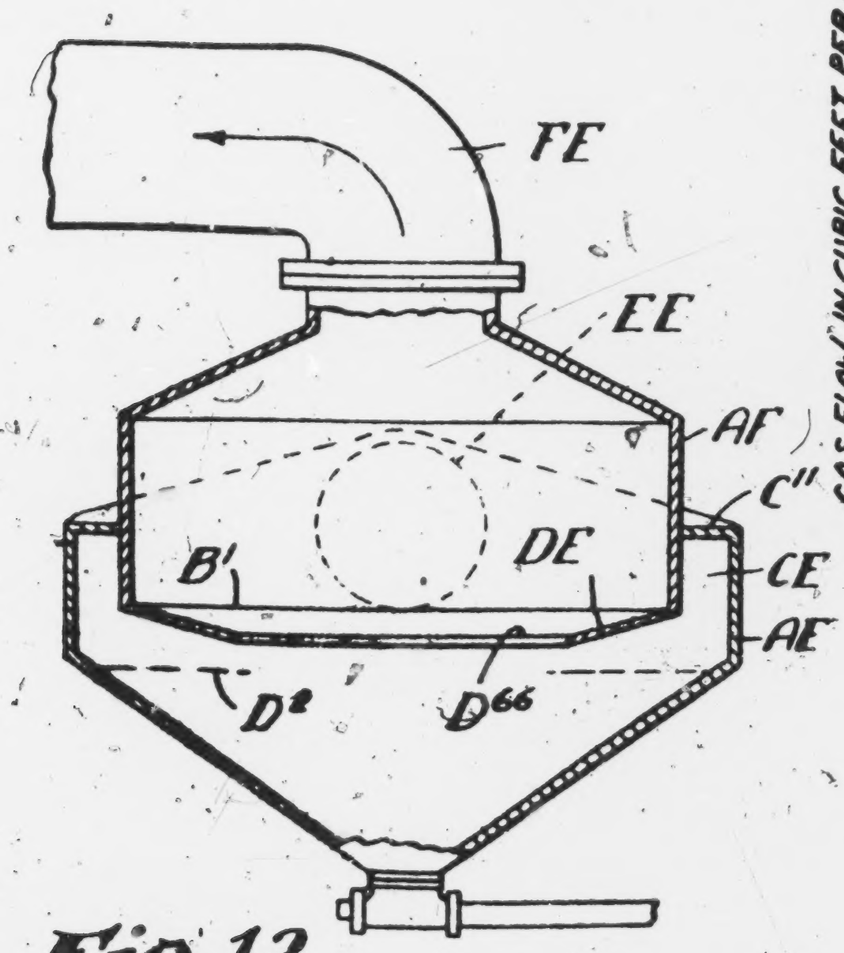


Fig. 12.

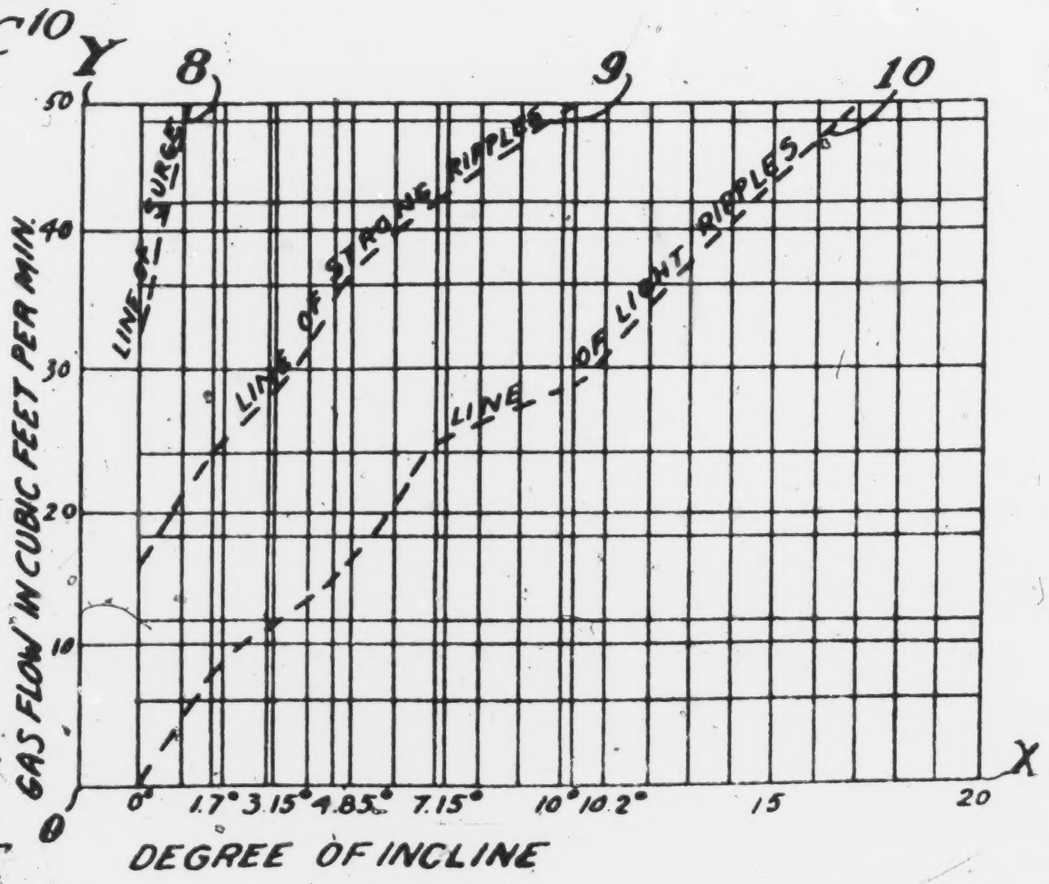
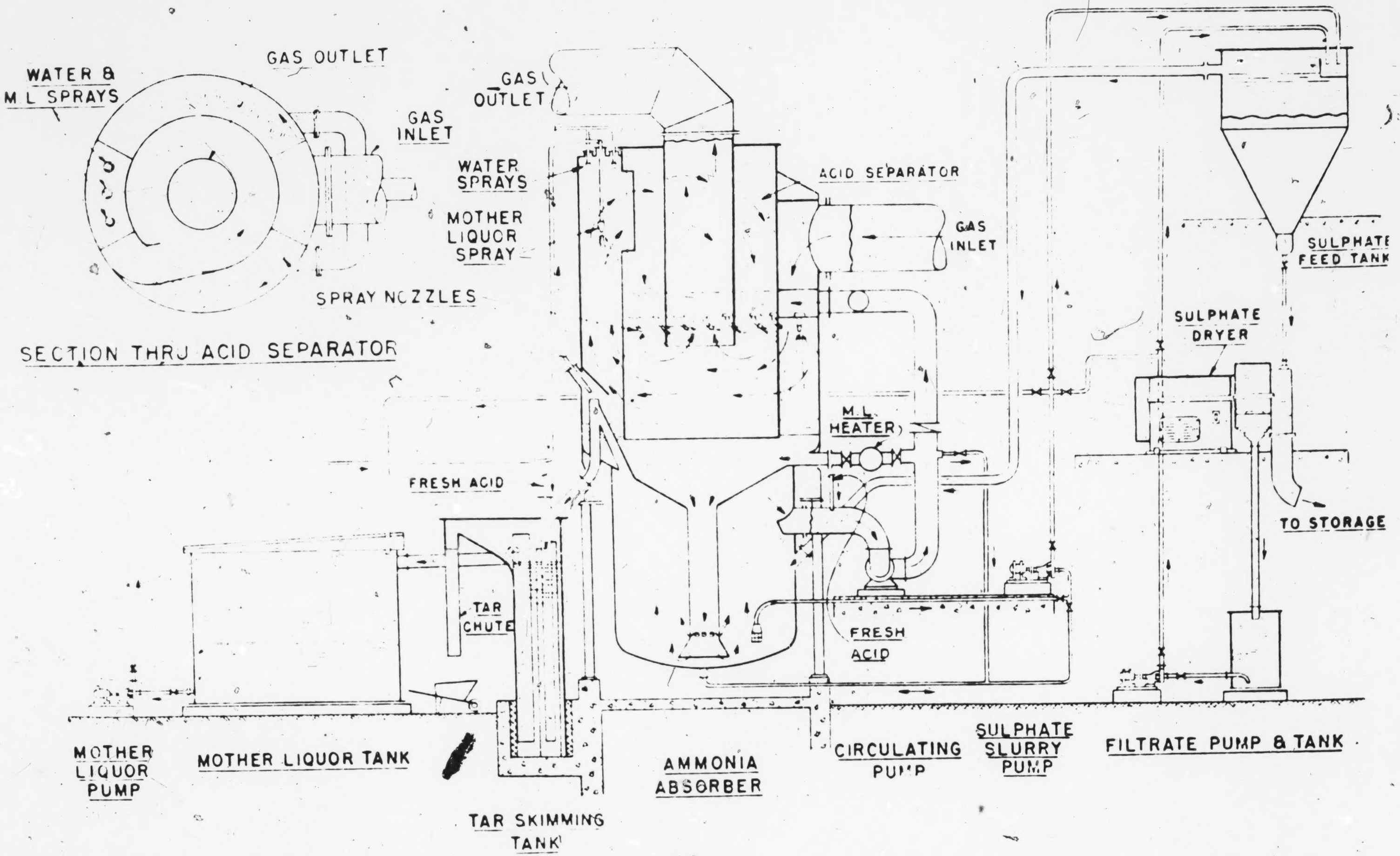


Fig. 8

INVENTOR
CARL OTTO
BY John E. MacDonald
ATTORNEY

KOPPERS AMMONIUM SULPHATE RECOVERY SYSTEM

PATENTS PENDING



p. 397 (1)

March 2, 1954 J. VAN ACKEREN 2,671,011

AMMONIUM SULFATE CRYSTAL PRODUCING APPARATUS

Filed Sept. 29, 1948 2 Sheets-Sheet 1

Fig. 2.

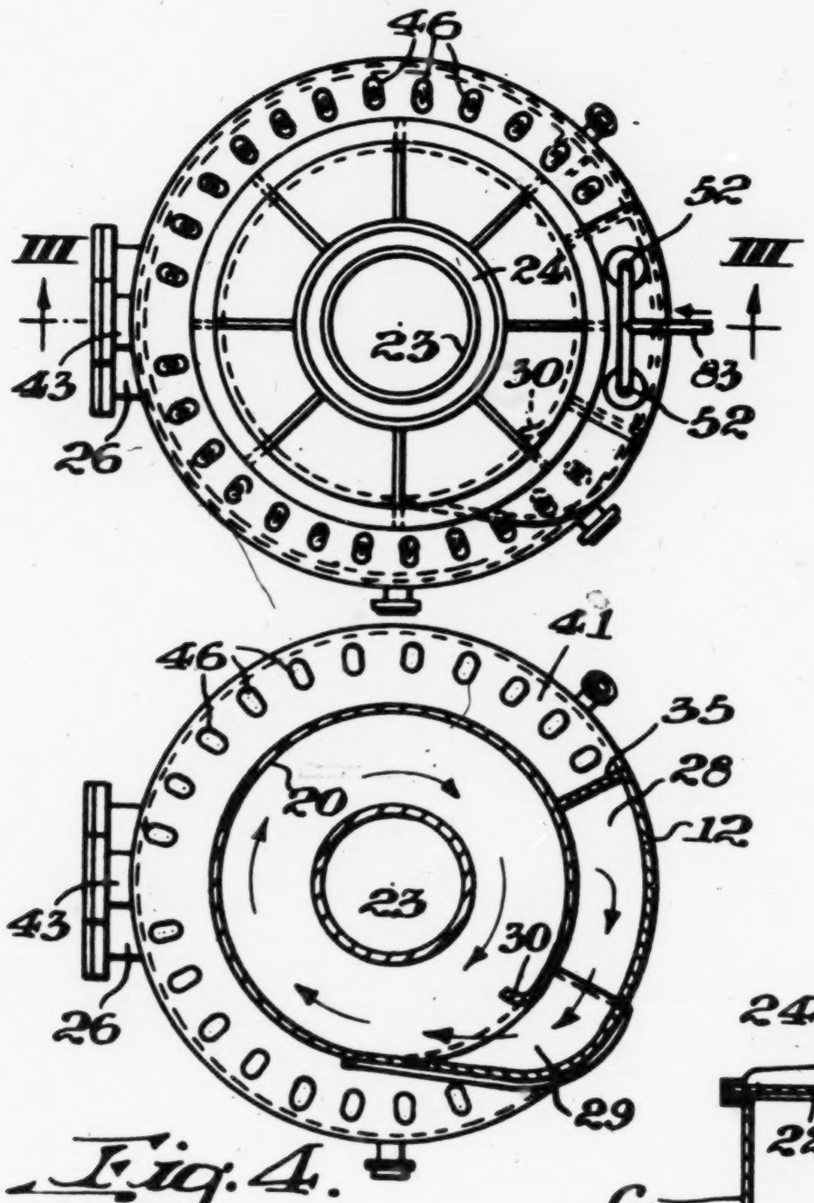


Fig. 4.

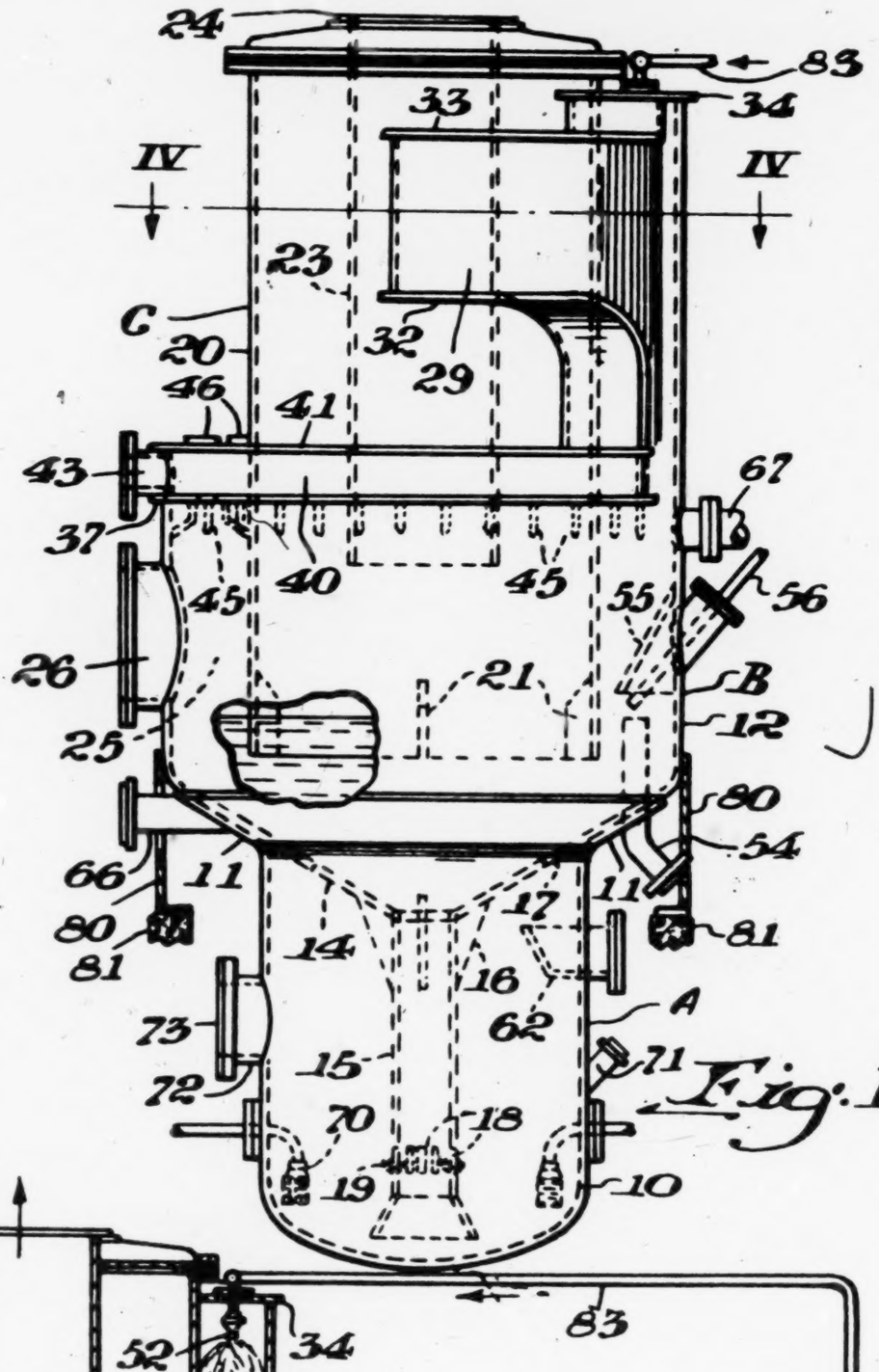
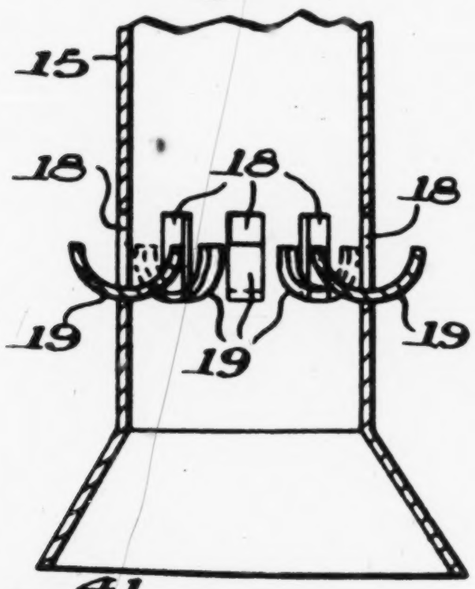


Fig. 1.

Fig. 5.



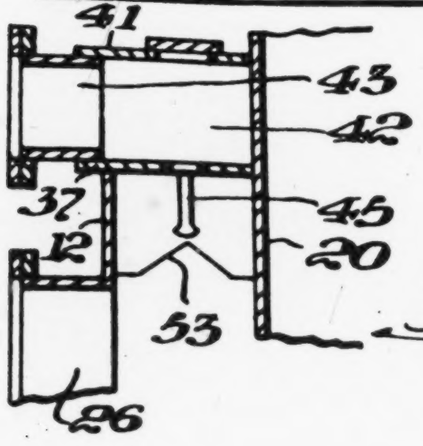


Fig. 8.

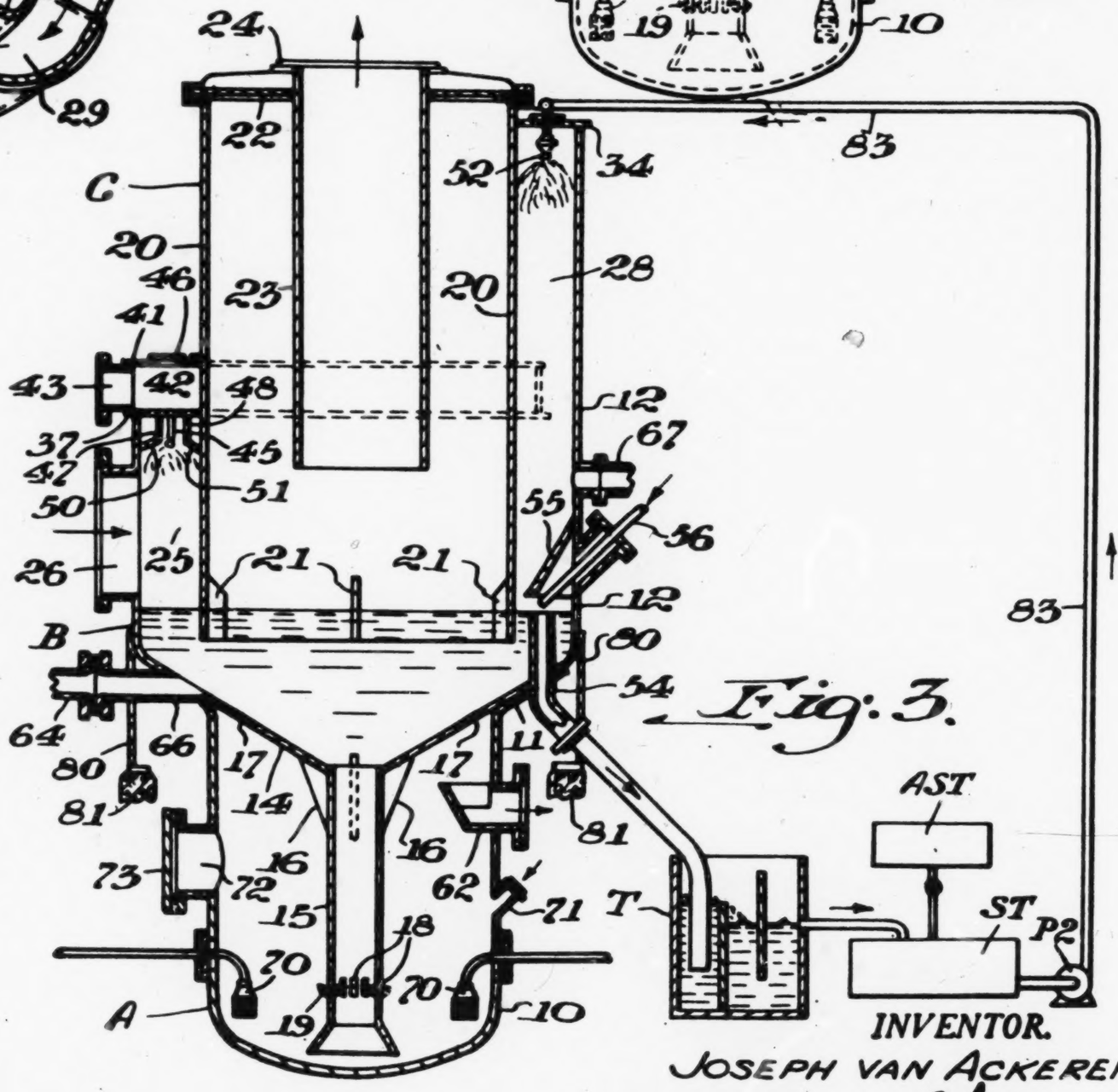


Fig. 3.

INVENTOR.
JOSEPH VAN ACKEREN.
BY Arthur L. Jerome
his ATTORNEY.

p. 397 (2)

March 2, 1954 J. VAN ACKEREN 2,671,011

AMMONIUM SULFATE CRYSTAL PRODUCING APPARATUS

Filed Sept. 29, 1948 2 Sheets-Sheet 2

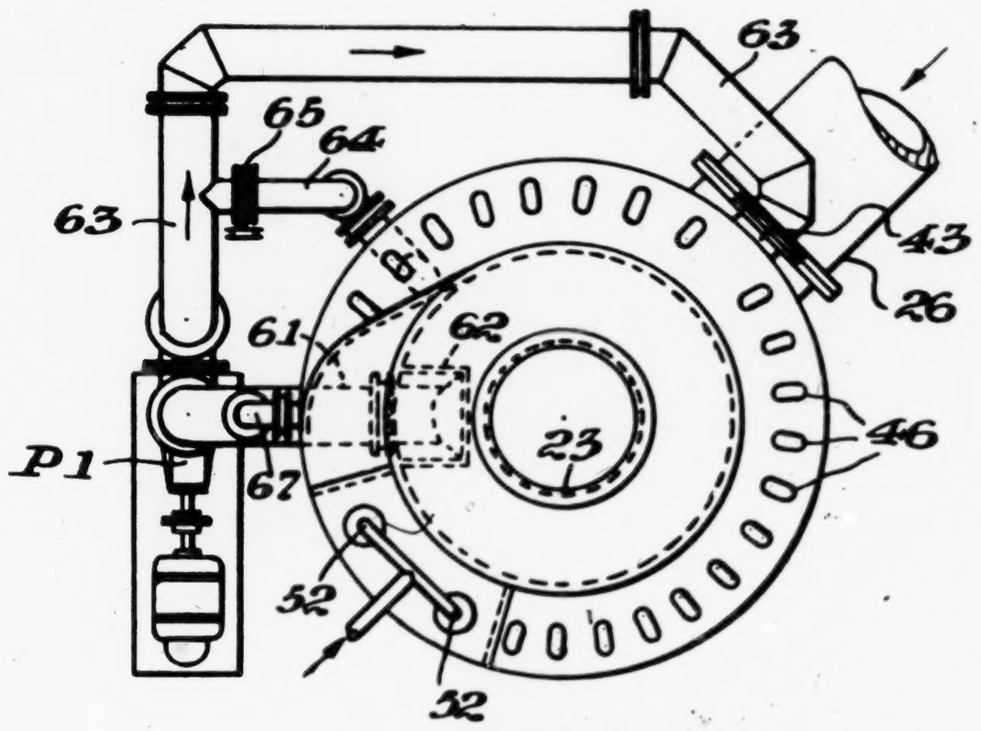


Fig: 6.

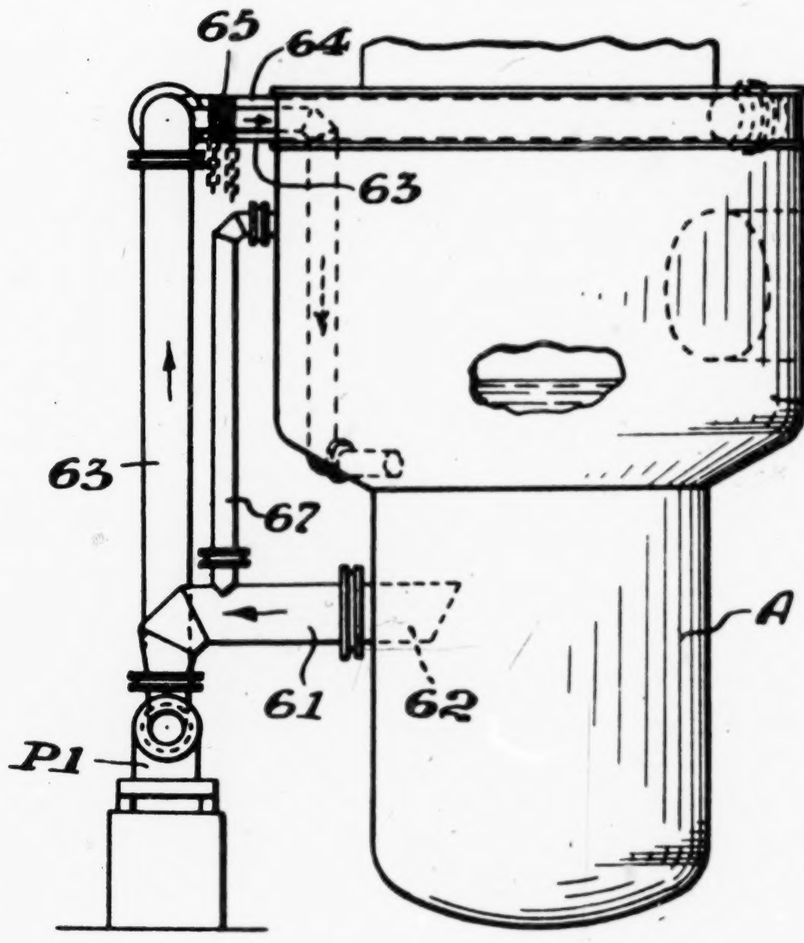


Fig. 7.

INVENTOR.
JOSEPH VAN ACKEREN.
BY Arthur L. Jerome
HIS ATTORNEY.

Plaintiff's Exhibit 8:

5. Auxiliary Equipment.

a. Liquor circulating pump.

1. 2500 G. P. M. at 15 lbs. per sq. in. head for 12'-0" spray type saturator.

2. Functions to circulate bath, absorb ammonia and build up crystal size.

d. Mother Liquor pump.

1. 3600 G. P. H. at 50 ft. head.

2. Circulates liquor from mother liquor tank through spray at saturator outlet to acid separator, thence from saturator bath overflow to tar skimmer to mother liquor tank.

3. Mother liquor being circulated is higher in acid than rest of bath because acid is added at tar skimmer.

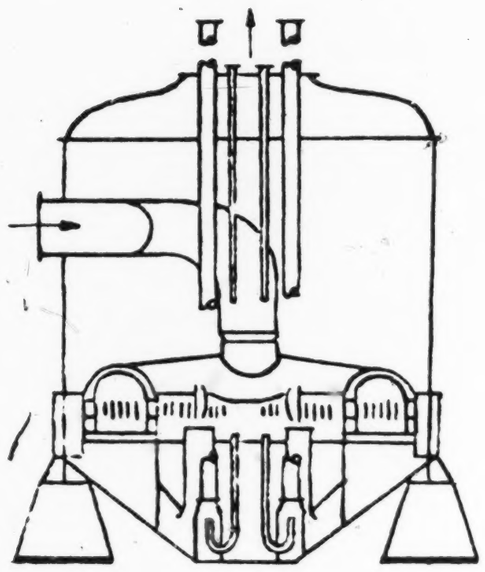
Drawings Attached to Plaintiff's Exhibit 8.

(See Opposite ☛)

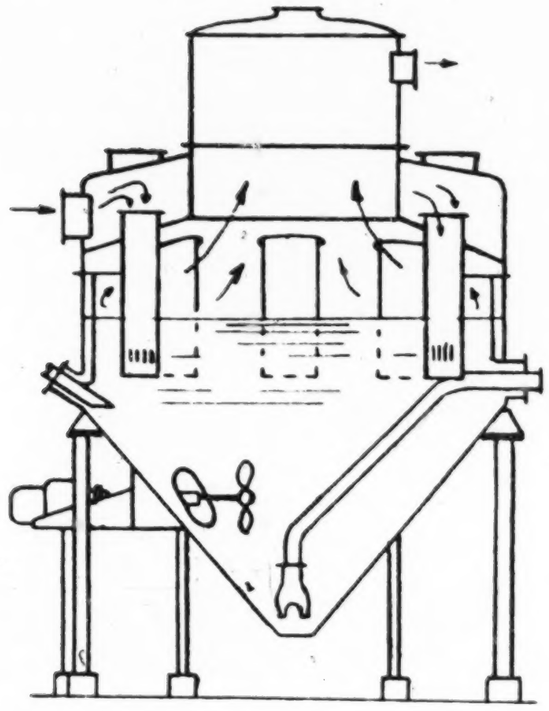
Note

Page 391 (sheet 1) is on card 10
Page 391 (sheets 2, 3) follow

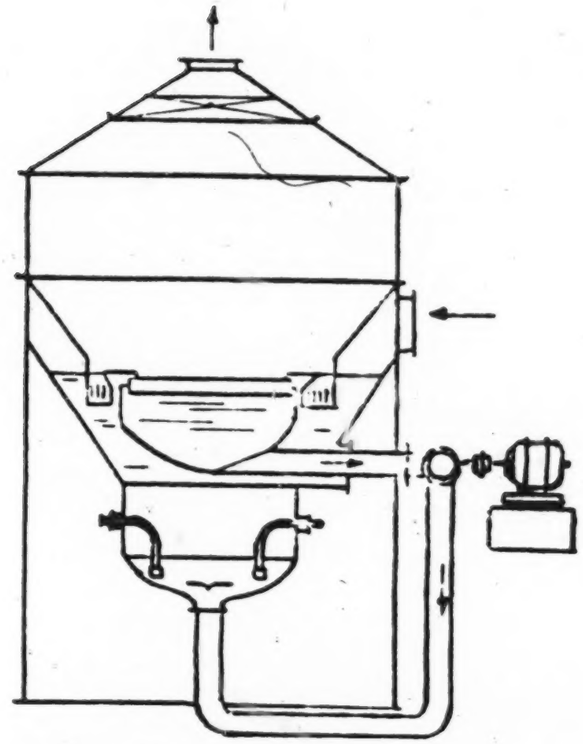
SATURATOR TYPES



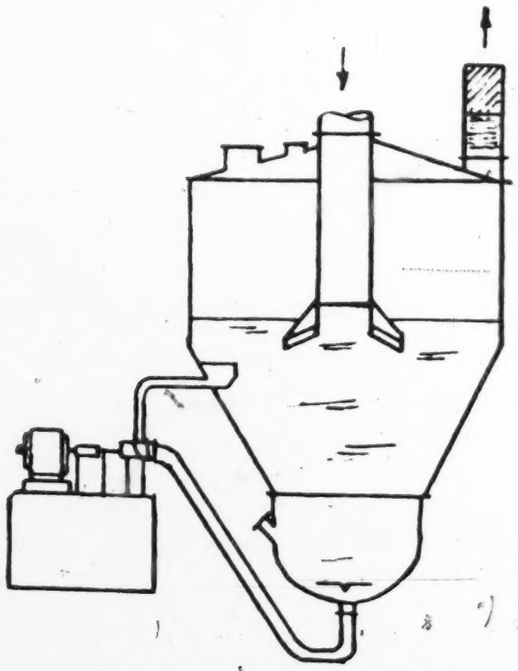
OLD CRACKER PIPE TYPE



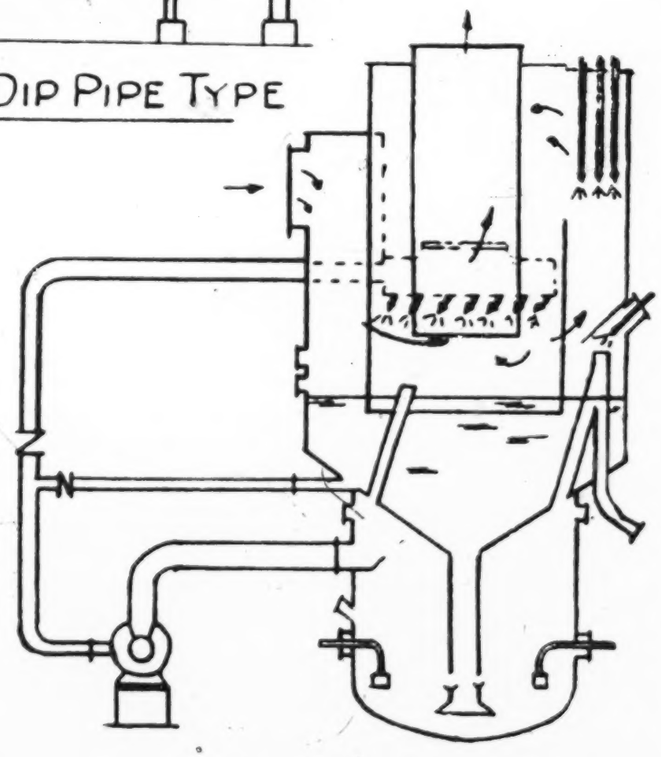
MULTIPLE DIP PIPE TYPE



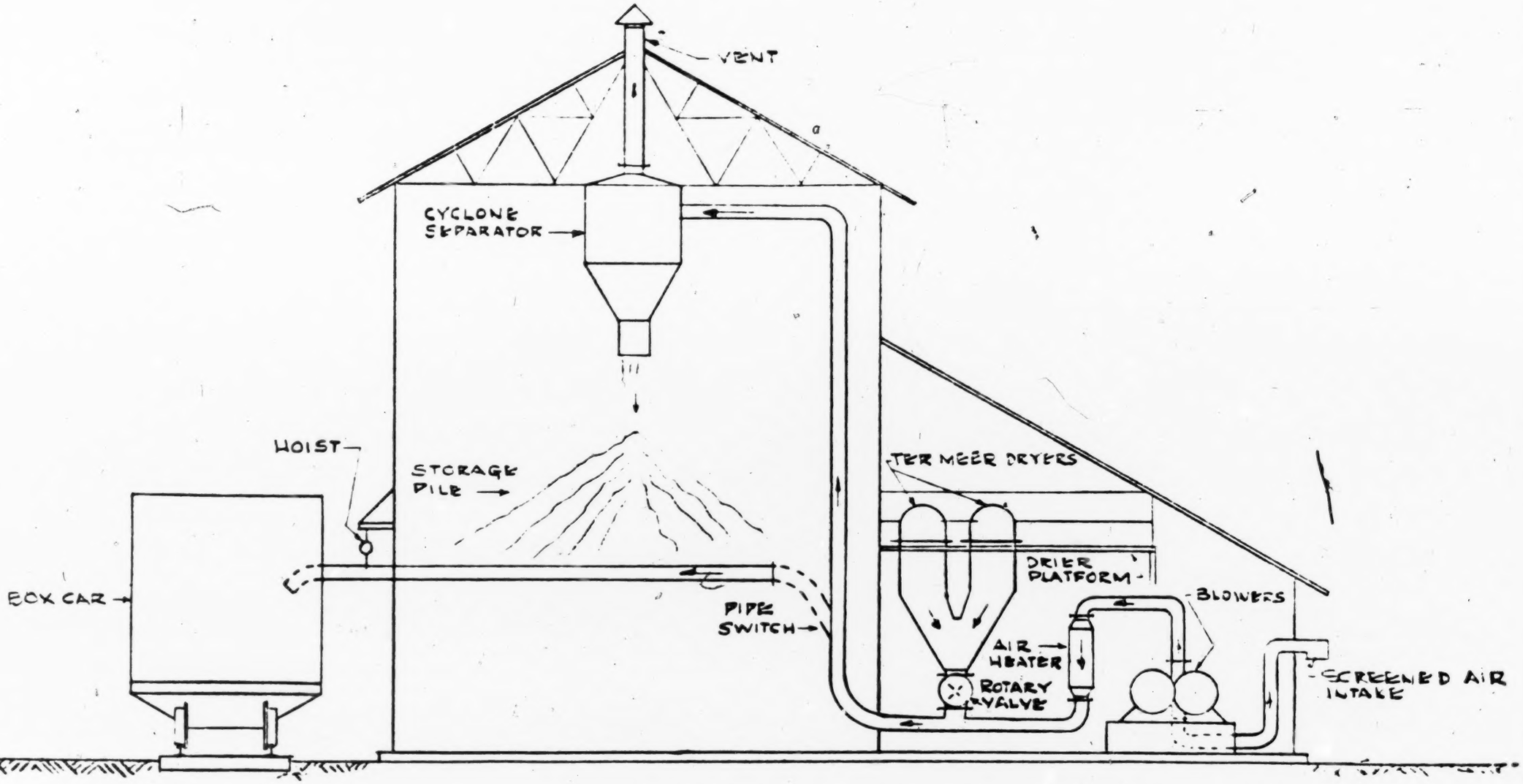
BUSTLE PIPE TYPE



CENTER DIP PIPE TYPE



SPRAY TYPE



PNEUMATIC SULPHATE HANDLING SYSTEM

Plaintiff's Exhibit 10.

STIPULATION

[Drawings Omitted]

It is stipulated by and between the attorneys for plaintiff and defendants that the following facts relate to the spray-type ammonia absorber which was installed by the Koppers Company, Inc. at the Neville Island plant of Pittsburgh Coke and Chemical Company.

1. The first sketches of the proposed absorber show Spraco nozzles, and illustrate the apparatus in a form very close to its final form. These sketches are dated Feb. 4, 1948, and carry the initials O. E. K. Photostats of these sketches are attached hereto and are marked Attachment A and Attachment B.

2. The first project drawings for this saturator all show Spraco nozzles. The earliest project drawing is P-3871-E dated Feb. 10, 1948, a copy of which is attached as Attachment C. A Spraco nozzle is indicated diagrammatically in the upper left-hand corner of this drawing.

3. In this saturator the gas flows horizontally through a curved channel which forms the primary absorption chamber, and the sprays project downwardly from the roof of this channel. Because of this horizontal gas flow it was at first considered advisable to provide baffle plates to prevent flow of gas above the cones of spray, which flow, if permitted to exist, would result in a portion of the gas by-passing the spray zone. An arrangement of this type is illustrated in project drawing P-3871-S, dated Mar. 19, 1948, a copy of which is attached as Attachment D. The baffle plates are colored green in this drawing.

4. After the contract No. 1790 was signed, detailed drawings were started. Spraco nozzles are called for in

drawing No. 2-B-417, dated May 17, 1948, a copy of which is attached as Attachment E.

5. Spraco nozzles for this saturator were ordered on Aug. 17, 1948 on Work letter 8648.

6. This order for Spraco nozzles was cancelled on Sept. 15, 1948 on Work letter 8653.

7. Splash-plate sprays are shown on drawing 2-A-319 dated Oct. 8, 1948, a copy of which is attached as Attachment F.

8. This change, made during the design stage, from Spraco nozzles to splash-plate nozzles was made to simplify the structure and reduce both operating and investment cost. It was felt that the splash-plate sprays would give better coverage of the horizontal gas channels. It was also felt that splash-plate sprays would cause less pressure drop, thus requiring less power for pumping the liquor.

9. First Run. The saturator, with splash-plate sprays as shown on drawing 2-A-319, Attachment F, was first run on Aug. 17-26, 1949. The ammonia removal was 94-96.5%. After this run the following changes were made.

(a) The "mother liquor" sprays were changed to Spraco nozzles.

(b) Baffle plates were added on each side of the main splash-plate sprays to reduce the amount of liquor striking the walls of the spray chamber.

10. Second Run. The saturator was given a second test run on Sept. 15-21, 1949, with ammonia removal of 97.5-99%.

Plaintiff's Exhibit 10.

11. On September 29, 1949 experiments were conducted by personnel of Pittsburgh Coke and Chemical Company with Spraco nozzles. These tests consisted of pumping water through a Spraco nozzle at several different pressures. Pictures of these tests are attached as follows:

Attachment G. Shows spray at 5 lbs. pressure.
Attachment H. Shows spray at 10 lbs. pressure.
Attachment I. Shows spray at 15 lbs. pressure.

12. After the second test run experimental work was conducted by Koppers at Swissvale on a model of the Neville Island Saturator. These tests showed that the splash-plate nozzles thoroughly covered the top of the spray chambers, but caused too large a proportion of the spray to strike the side walls, with inadequate coverage of the lower portion of the spray chambers. The tests also showed that Spraco nozzles provided a much better distribution of the spray over the main portion of the spray chamber, but left a void space above the cone of spray, as had been expected.

Further tests showed that the void spaces above the Spraco sprays could be fairly well covered by drilling holes in the nozzles in such a manner that small streams of liquor were projected upwardly at an angle against the sides and top of the spray chamber.

13. As the result of these experiments the following changes were made:

(a) 12 of the splash-plate sprays were replaced by Spraco No. 15a nozzles with holes drilled as described above.

(b) 8 of the splash-plate sprays, modified by adding deflector plates to confine the spray laterally, were left in use.

(c) The Spraco nozzles at the "mother liquor" sprays were replaced by smaller Spraco nozzles.

(d) The "mother liquor" pumps were connected in series to provide the higher pressure required by the smaller spray nozzles.

14. A Third Run was made from Oct. 5 to Dec. 3, 1949 (date of shut-down not known precisely, but occurred between Dec. 3, 1949 and Jan. 27, 1950), with ammonia removal of 99.75-99.92%.

During this run the original liquor circulating pump was used, providing a pressure of approximately 5 psi on the spray header.

At this time the saturator was operating satisfactorily, and the Koppers men left the plant.

15. Mr. Schreiber, of Pittsburgh Coke and Chemical Co., wanted to provide leeway to permit production of ammonia sulphate from large quantities of *anhydrous ammonia* at the same time that ammonia was being absorbed from coke oven gas.

To test out this procedure, large quantities of *anhydrous ammonia* were added to the absorber during part of the 3rd Run. There was no adverse effect on the ammonia content of the outlet gas, thus indicating that no change in the apparatus was required to make this type of operation possible.

16. However, Koppers had agreed, prior to the 3rd Run, to make some additional changes. These changes were subsequently made:

(a) All of the nozzles in the primary absorption chambers were replaced with stainless steel Spraco nozzles, size 13a, tilted at an angle to both hori-

zontal and vertical. It has been demonstrated at Swissvale that by this arrangement the Spraco nozzles would fill the entire annular spray chamber with spray. This arrangement is shown on drawing 1790-2A5, a copy of which is attached as Attachment J.

(b) A new liquor circulating pump providing a pressure of 17 psi on the spray header was installed.

17. The changes mentioned in Par. 16 above, along with other changes, were approved in Deviation No. 10, a copy of which is attached as Attachment K.

February 20, 1956.

Plaintiff's Exhibit 27.

(See Opposite ☛)

Patented Mar. 2, 1954

2,671,011

P. 347 (3)

UNITED STATES PATENT OFFICE

2,671,011

AMMONIUM SULFATE CRYSTAL PRODUCING APPARATUS

Joseph van Ackeren, Pittsburgh, Pa., assignor to Koppers Co., Inc., Pittsburgh, Pa., a corporation of Delaware

Application September 29, 1948, Serial No. 51,724

6 Claims. (Cl. 23—273)

1

This invention relates to improved apparatus and to an improved method for treating gases, such as coke oven gas, to remove ammonia and similar constituents therefrom.

It is customary in the art to recover the ammonia in coke oven gas by passing the gas through a saturator containing a weak sulfuric acid solution through which the gas is bubbled with the result that the ammonia in the gas, and the sulfate radical in the acid solution, combine to form sulfate of ammonia which crystallizes out of the acid solution.

In order to obtain substantially complete removal of the ammonia from a gaseous mixture, it is necessary not only for all of the gas to come into contact with the sulfuric acid bath, but to come into contact with an acidic portion of the bath capable of reacting with ammonia. To secure adequate and effective contact between the gas and the liquid of the sulfuric acid bath, it has been customary to introduce the gas into the liquid of the bath at a point a substantial distance below the surface of the liquid and permit the gas to bubble upwardly through the liquid. An inherent disadvantage of this arrangement is the relatively high pressure required to force the gas below the surface of the liquid, and the relatively high cost of operating the compressors or blowers for developing this pressure.

In the processes heretofore employed the gas leaving the saturator carries with it particles of acid, and it has been customary to provide, as a separate piece of apparatus, an acid separator for removing the acid from the gas leaving the saturator and returning this acid to the saturator.

An object of this invention is to provide an improved method and apparatus of the type described which eliminates the necessity for maintaining such pressure on the gaseous mixture as is required when it is introduced into the acid bath in the saturator at a point below the level of the surface of the bath.

Another object of the invention is to provide an improved method and apparatus of the type described which eliminates the necessity for an acid separator as a separate piece of equipment.

A further object of the invention is to provide an improved saturator which incorporates therein an acid separator.

Another object of the invention is to provide an improved saturator in which there is no liquid seal to create back pressure to oppose the flow of the gaseous mixture through the saturator (i. e. the flow of gas is unobstructed in the sense that there is no head of liquid opposing its flow).

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A further object of the invention is to provide an improved saturator in which the gaseous mixture is passed through an acid spray, and in which the direction of flow of the gaseous mixture is continuously altered to thereby create turbulence and insure intimate and complete admixture of the gas and of the acid as the gas passes through the acid spray.

Another object of the invention is to provide an improved saturator of the type described and in which a structural element, which is a part of the duct forming a torturous path for the gas entering the saturator, is also a part of the cyclone separator for removing acid from the gas leaving the saturator.

A further object of the invention is to provide an improved saturator of the type described which is arranged so that a large volume of liquid may be circulated with a minimum of expense for pumping.

Another object of the invention is to provide an improved saturator of the type described which is arranged so that the vertical distance through which liquid of the acid bath must be elevated is kept at the minimum to thereby reduce the expense of pumping the liquid.

A further object of the invention is to provide an improved saturator of the type described which incorporates means for insuring efficient and substantially complete removal of the pyridine and related constituents from the gas passing through the saturator.

Another object of the invention is to provide an improved saturator of the type described which is arranged to insure the production of ammonium sulfate crystals of controllable size.

A further object of the invention is to provide a saturator incorporating improved means for insuring that movement of supersaturated solution upward through a bed of crystals in the saturator will be at a substantially uniform rate throughout the entire area of the crystal bed.

Other objects of the invention and features of novelty will be apparent from the following description taken in connection with the accompanying drawings in which:

Fig. 1 is a side elevation of my improved saturator, with a part broken away to show the interior construction;

Fig. 2 is a top or plan view of the saturator shown in Fig. 1;

Fig. 3 is a sectional view of the saturator and its associated apparatus taken on the line III—III of Fig. 2;

Fig. 4 is a sectional view taken on the line IV—IV of Fig. 1;

Fig. 5 is a fragmentary enlarged view of the lower end of the slurry-circulating pipe and showing the slurry deflectors and by-pass passages;

Fig. 6 is a top view of the saturator together with the associated piping and liquor-circulating pump;

Fig. 7 is a fragmentary elevational view showing the lower portion of the saturator and the associated piping, with the supporting skirt omitted; and

Fig. 8 is a fragmentary enlarged sectional view showing a modification which I may employ.

In Figs. 6 and 7 of the drawings, in order to more clearly illustrate the arrangement of the apparatus, some of the pipe connections to the saturator are shown in somewhat different rotative positions than they are shown in the other figures of the drawings.

Referring to the drawings, it will be seen that the saturator is generally cylindrical in form, and has a lower, or crystallizing bath-containing portion A, a central or absorbing portion B, and an upper or acid-separator hood portion C.

The saturator is preferably constructed of sheet stainless steel of a type which is resistant to the action of acid of the concentrations customarily employed. The various individual sections are welded together with suitable stainless steel welding rod to provide a gas and liquid tight structure, and the welded areas are thereafter ground and polished to present smooth surfaces which offer little opportunity for the adherence of crystals. Hence, the tendency for salt incrustations to develop in the saturator is held to the minimum.

The lower or bath-containing portion A of the saturator is a substantially cylindrical sheet metal bowl 10 which is closed at the bottom by a curved head. The upper edge of this bowl flares radially outwardly, as indicated at 11, and is secured to the lower face of the bottom wall of the central or absorber portion B of the saturator.

The central or absorber portion B of the saturator is a vessel having a cylindrical outer wall 12 which is of somewhat larger diameter than the lower portion A of the saturator. The outer wall 12 is secured to a conical, dished head 14, the surface of which slopes downwardly to a central slurry circulating pipe 15 of relatively large diameter.

A plurality of angular braces, or reinforcing members 16, are secured to the outer surface of the pipe 15 and to the bottom surface of the head 14 of the absorber portion B to accurately and rigidly secure the pipe 15 to the head 14.

A plurality of holes 17 in the head 14 near the point of attachment of the bowl 10 with the head 14 prevent entrapment of gas in the upper part of the crystallizing portion of the saturator. The holes 17 are of such size, and their flow capacity is so limited, that the preferred hereinafter-described circulation of acid solution throughout the saturator is not materially affected by flow of solution through these holes.

The pipe 15 is of such length, as is clearly shown in Figs. 1 and 3, as to extend to a point a short distance above the bottom of the lower or crystallizing bath-containing portion A. The lower end of the pipe 15 is flared or enlarged somewhat to reduce the rate of flow of liquid in this portion of the pipe. In addition, as is best shown in Fig. 5, the lower end of the pipe has a plurality of rectangular openings 18 therein,

and a curved deflector 19 is secured in each of these openings.

As hereinafter explained, a portion of the liquid flowing downwardly in the pipe 15 is diverted by the deflectors 19 through the openings 18 and thence upwardly in the crystallizer bowl.

The upper or acid-separator hood portion C includes a substantially cylindrical member 20 of somewhat smaller diameter than the wall 12 of the absorber portion B of the saturator. The member 20 is located centrally or coaxially of the wall 12 of the absorber portion, and, as is best shown in Fig. 3, the member 20 extends downwardly within the wall 12 to a point a short distance above the head or lower wall 14 of the absorber portion B. The lower end of the cylindrical member 20 is open, while a plurality of inwardly extending ribs 21 are secured on the inner face of the member 20 and prevent swirling or circular motion of the liquid in the saturator.

The upper end of the cylindrical member 20 is closed by a head 22 through which extends a centrally located pipe 23, Fig. 3, of relatively large diameter. The pipe 23 extends downwardly from the head 22 a substantial distance, the lower end of the pipe 23 being open and being located some distance above the lower edge of the member 20. The upper end of the pipe 23 is surrounded by a flange 24 and serves as the outlet for gas leaving the saturator.

As explained above, the cylindrical hood member 20 and the cylindrical wall 12 are substantially concentric, and the member 20 extends within the wall 12. Hence, an arcuate or curved channel passageway or absorption chamber 25 is formed between the members 12 and 20, the outer wall of the chamber being formed by the wall 12, and the inner wall of the chamber being formed by the member 20.

As can be seen from the above description and from the drawings, passageway or chamber 25 defines a relatively narrow elongated absorption chamber or zone whose use and operation are described more fully below.

The wall 12, together with the lower wall or head 14, form a vessel which is adapted to hold a body of solution, and the cylindrical member 20, together with the head 22, form an inverted vessel which helps to close the top of the absorber portion B as well as to function as an acid separator. The member 20 and the associated elements may also be considered as a conduit through which gas leaving the saturator passes.

A relatively large opening 26, Fig. 1, is provided in the wall 12 and serves as an inlet for the flow of gas into the channel absorption chamber 25 and thus into the saturator.

A portion of the chamber 25 diametrically opposite from the inlet opening 26 communicates with a vertically extending passage 28, Figs. 3 and 4, the inner wall of which is formed by the cylindrical member 20 and the outer wall of which is formed by an upward extension of the wall 12. Hence, the radial extent of the passage 28 is the same as the chamber 25, while the circumferential extent of the passage 28 is such that the cross-sectional area of this passage is approximately the same as that of the inlet opening 26.

The upper end of the vertical passage 28 communicates with a horizontal passage 29, Figs. 1 and 4, which extends a portion of the distance around the cylindrical member 20. As is best shown in Fig. 4, the cylindrical member 20 has an opening therein into which the passage 29 connects, the outer wall of the passage 29 being

curved adjacent the vertical passage 28 and gradually merging into a substantially straight portion which is tangent to the cylindrical member 26. The portion of the cylindrical member 26 on the side of the opening in this member adjacent the vertical passage 28 is curved inward slightly, as indicated at 30.

The lower wall of the horizontal passage 29 is formed by a curved plate 32, Fig. 1, which also forms one side wall of the vertical passage 28. The upper wall of the horizontal passage 29 is formed by a plate 33, one end of which curves upwardly and is joined to a plate 34, which closes the upper end of the vertical passage 28. The other side wall of the vertical passage 28 is formed by a plate 35, Fig. 4.

The upper face of the chamber 25 is closed by an arcuate plate 37, Fig. 3, which extends circumferentially around the saturator from the plate 35 at one side of the vertical passage 28 to the plate 32 at the other side of the passage 28. The inner edge of the plate 37 is secured to the outer face of the cylindrical member 26, while the outer edge of the plate 37 is secured to the top of the wall 12.

A curved plate 40, Figs. 1 and 2, is secured on the upper face of the plate 37 near its outer edge and extends around the saturator from the plate 32 on one side of the vertical passage 28 to the plate 35 on the other side of the vertical passage 28.

An arcuate plate 41, Figs. 1, 2, 3, and 4, is secured to the upper edge of the plate 40 and extends to and is secured to the outer face of the cylindrical member 26. The plate 41 extends around the saturator from the plate 32 at one side of the vertical passage 28 to the plate 35 at the other side of this passage.

The plates 37, 40 and 41 and the cylindrical member 26 cooperate to form an arcuate duct 42, Figs. 3 and 4, extending around the saturator except where the vertical passage 28 is located. An inlet 43 to the duct 42 is provided at a point substantially opposite the vertical passage 28 so that the nearly semi-circular portions of the duct on opposite sides of the inlet 43 are substantially equal in length.

A plurality of downwardly directed nozzles 45, Figs. 1 and 3, are secured in the plate 37 and are located substantially intermediate the walls 12 and 26 so that the spray from the nozzles fills the channel absorption chamber 25. The nozzles 45 may be secured in the plate 37 in any desired manner, while the plate 41 has openings therein directly above the nozzles 45 to permit ready access to the nozzles. The openings in the plate 41 are closed by suitable cover plates 46, Figs. 1, 2, 3 and 4. The duct 42 forms a manifold or header for supplying acid solution to the nozzles 45.

The nozzles 45 extend downwardly from the plate 37 a substantial distance, and means is provided to prevent circulation of gas in the portion of the chamber 25 above the tips of the nozzles. As shown in Fig. 3, plates 47 and 48 extend downwardly from the plate 37 on opposite sides of the nozzles 45. The plates 47 and 48 are located relatively near the nozzles 45 so there is little space for flow of gas between these plates and the nozzles.

The plates 47 and 48 are curved, as viewed from the top of the saturator, and extend throughout the chamber 25. A conical plate 50 extends from the plate 47 to member 12, while a similar plate 51 extends from the plate 48 to the cylindrical member 26. The plates 47, 48, 50 and 51, therefore, enclose the area above the tips of the nozzles 45 and substantially exclude gas from flowing in the region above the spray from the nozzles. This prevents gas from passing through the saturator without being exposed to spray of solution from the nozzles, and it inhibits accumulation of salt on the surfaces above the nozzles.

In Fig. 8 of the drawings there is shown a different arrangement for preventing flow of gas in the area above the tips of the nozzles. In this modification a plurality of radially extending plates 53 are provided. These plates extend from the outer face of the cylindrical member 26 to the inner face of the wall 12, and the upper edge of each of these plates is secured to the lower face of the plate 37. The lower side of each of the plates 53 has therein a centrally located triangular notch which is of such size and location that the spray from adjacent nozzles 45 fills the area of the notch. The plates 53 effectively prevent flow of gas in the area above the spray from the nozzles 45, and insure that all of the gas flowing through the chamber 25 comes into contact with spray from the nozzles 45.

Two nozzles 52, Figs. 2, 3 and 6, are mounted in the top plate 34 at the upper end of the passage 28. The nozzles 52 spray liquid into the gas as it passes upwardly in the passage 28. As hereinafter explained, the liquid for the nozzles 52 is preferably supplied from a different source than the liquid supplied to the nozzles 45.

This saturator includes a liquid level control means for maintaining the liquid in the saturator at a selected level. As illustrated this liquid level control means comprises an overflow pipe 54, Fig. 3, which projects through the wall 14 and extends upwardly to a point a substantial distance, such as 10 inches, above the bottom of the cylindrical member 26. The overflow pipe 54 determines the level of the liquid in the saturator and this is fixed at a selected level high enough above the bottom of the member 26 to prevent gas from the chamber 25 from flowing under the bottom of the cylindrical member 26 and thence to the outlet pipe 23 without passing through the vertical passage 28, and through the separator portion of the saturator. The overflow pipe 54 communicates with the usual tar-skimmer tank T.

A hood 55, Fig. 3, is secured to the inner face of the wall 12 and covers the opening into the pipe 54 so that spray from the nozzles 45 and 52 will not flow directly into the overflow pipe 54. The upper edge of the hood 55 is secured to the wall 12 while the lower edge of the hood extends inwardly beyond the pipe 54, and a short distance, such as 4 inches, above the pipe 54. The hood 55 extends at a relatively steep angle so liquid will flow off it rapidly and thus prevent the deposit of crystals on this member.

A pipe 56, Figs. 3 and 4, communicates with the interior of the saturator at a point beneath the hood 55. The pipe 56 may be employed to spray hot water on the overflow pipe 54 and thus prevent clogging of this pipe by tar which is carried into the saturator by the gas.

The saturator has associated therewith a pump for circulating liquor in the lower or bath-containing portion of the saturator and for supplying liquor to the nozzles 45. Referring to Figs. 6 and 7, it will be seen that a motor driven pump P1 is provided and that the inlet of this pump communicates with the lower or bath-containing portion A of the saturator by a pipe 61. The pipe 61 opens into the lower portion A of the saturator through a suction head 62, which is a substan-

tially rectangular bowl-shaped member, which is open only at the top. The side walls of the member 62 flare outwardly, as shown in Fig. 6, so that the opening into the suction head is of relatively large area with the result that the velocity of the liquid flowing into the suction head is correspondingly low. The upper edge of the side walls of the suction head 62 is in substantially the same horizontal plane throughout the periphery of this member so that the flow of liquid into the suction head is at a substantially uniform rate at all points. As is obvious in Figs. 1 and 3 of the drawings, the suction head 62 is located near the top of the lower portion A of the saturator.

The pump P1 supplies liquid through a pipe 63 to the duct 42, Fig. 3, and thus to the nozzles 45. The duct 42, therefore, serves as a manifold or header for supplying liquid to the nozzles 45. A branch pipe 64, Figs. 6 and 7, governed by a valve 65, leads from the pipe 63 to a connection 66 which opens into the saturator at a point below the level of the liquid in the saturator. The by-pass pipe 64 and the valve 65 enable the circulation of liquid in the saturator to be increased by a variable amount in excess of that provided by the flow of liquid through the nozzles 45.

A pipe 67, Fig. 7, connects to the inlet pipe 61 at a point between the pump P1 and the saturator and leads to the interior of the saturator at a point above the level of the liquid in the saturator. The pipe 67 is a vent and provides means for the escape of gases from the inlet pipe 61.

Suction heads 70, Figs. 1 and 3, of conventional construction, extend into the lower portion of the bowl 10 and provide means for withdrawing ammonium sulfate crystals from the saturator and for supplying the sulfate to a centrifugal dryer, not shown. Liquid removed from the crystals in the dryer is returned to the saturator through the pipe connection 71.

A relatively large opening 72, Figs. 1 and 3, is provided in the bowl 10 and is closed by a cover 73. This opening enables workmen to secure access to the interior of the lower portion of the saturator during its construction, or for maintenance purposes, after it has been in use.

A skirt 80, Figs. 1 and 3, surrounds the saturator and is secured to the exterior of the member 12. The skirt 80 extends downwardly and rests upon suitable supporting means, such as the reinforced concrete abutments 81, to provide a support for the saturator.

Liquid from the tar tank T overflows into a storage tank ST, from which it is pumped by a suitable pump P2 to a pipe 83, Figs. 1, 2 and 3, and thus to the nozzles 52. Make-up acid from an acid storage tank AST is also supplied to the storage tank ST, and thus is supplied through pipe 83 to the nozzles 52. The amount of make-up acid supplied from the acid storage tank AST to the tank ST is regulated so that the liquid discharged from the nozzles 52 has an acid content about twice as high as the liquid in the saturator bath, that is, about 11% acid in the spray from the nozzles 52, as compared to about 5½% acid in the saturator bath.

Make-up acid, in addition to that supplied through the nozzles 52, may be supplied to the saturator in any desired manner.

Under normal operating conditions the bath level in the saturator is maintained approximately constant by the overflow pipe 54, while liquid is withdrawn from the upper part of the bowl 10 by the pump P1 through the suction head 62, and is supplied to the header or manifold 42 from which it flows through the nozzles 45 and is sprayed throughout the absorption chamber 25 and falls to the surface of the bath at the bottom of this chamber. In addition, liquid supplied by pump P1 through the by-pass pipe 64 is added to the bath at the bottom of chamber 25.

Similarly, the liquid discharged from the nozzles 52 at the upper end of the vertical passage 28 is sprayed throughout the passage 28 and falls to the surface of the liquid in the saturator.

Likewise, liquid which collects on the interior of the wall 20 of the acid-separator portion C of the saturator flows down this wall and adds to the liquor in the saturator bath. It will be seen that the arrangement for the return of liquid from the acid-separator to the saturator bath is such that it is unnecessary for this liquid to flow through small pipes which may become clogged by salt deposits. The incorporation of the acid separator in the saturator, therefore, eliminates a troublesome operating difficulty which exists when the acid separator is an independent unit.

The liquid in the portion of the saturator above the header or division wall 14 flows downwardly through the pipe 15 to the lower or bath-containing portion of the saturator to replace the liquid removed by the pump P1.

Coke oven, or other gas to be processed, is supplied to the saturator through the inlet opening 26, Fig. 1 and 3, to the chamber 25 and the vertical passage 28. The vertical passage 28 is diametrically opposite the inlet opening 26 while the channel 25 which is located radially outwardly of the cylinder member 20 provides two paths in multiple through which gas may flow from the inlet opening 26 to the vertical passage 28. As viewed in Figures 2 and 3 of the drawings one of the channels extends from the inlet opening 26 in a clockwise direction around the member 20, while the other channel extends from the inlet opening 26 in a counterclockwise direction around the member 20. Both of these channels communicate with the lower end of the passage 28.

As the gas passes through the chamber 25, it must pass through spray from the nozzles 45 which, as can be seen from the drawing are uniformly distributed over the entire length of absorption zones 25. This spray is extremely dense and completely fills the chamber 25. The rate of flow of gas through the chamber 25 is relatively high so that the gas is extremely turbulent. Furthermore, because of the curvature of the two portions of the chamber 25 through which the gas flows, the gas in flowing through these portions is constantly changing direction and there is no laminar flow of gas or stratification of the gas. Due to the extended gas-liquid interface existing in the mixture of spray and gas, the absorption of ammonia proceeds rapidly to completion and by the time the gas reaches the vertical passage 28, it is substantially free of ammonia. The turbulent flow of gas through the channels 25 is also very effective in bringing the gas into contact with the acid absorbing liquid which forms the bottom walls of the channels.

As just explained, the gas supplied through the inlet passage 26 divides into two streams flowing horizontally in curvilinear paths, one of which flows in one direction through a substantially semi-circular portion of chamber 25 while the other gas stream flows through the other portion of the chamber 25. Hence, there are two arcuate paths in multiple between the inlet opening 26 and the vertical passage 28, and each of these

paths is required to handle only one-half of the gas. Hence, each path can be of smaller cross-sectional area than would be necessary if each path was required to handle the entire volume of gas. As a result, the chamber 25 may be made of substantially smaller size and of lesser vertical extent than would otherwise be required. The reduced vertical extent of the chamber 25 reduces the vertical distance through which liquid must be pumped to the nozzles 45, so the pumping cost is reduced to the minimum.

The gas streams flowing through the two portions of the chamber 25 in multiple meet at the base of the vertical passage 28 and flow upwardly through this passage in countercurrent to the liquid sprayed from the nozzles 52 at the top of this passage.

As previously explained, the liquid sprayed from the nozzles 52 has a substantially higher acid content than that supplied from the nozzles 45. This liquid of high acid content has a greater affinity for pyridine and its congeners than the liquid of low acid content, and, therefore, removes the gas passing through the vertical passage 28, any of these constituents not removed by passage of the gas through the chamber 25.

The liquid from the nozzles 52, together with the pyridine and other constituents absorbed thereby, adds to the bath in the saturator and increases the pyridine concentration of this bath. The pyridine may be removed from the bath by any suitable means well known in the art so as to maintain the pyridine concentration of the saturator bath at a satisfactorily low level to thereby make possible substantially complete recovery of the pyridine.

When the gas reaches the upper part of the vertical passage 28, the gas flows through the horizontal passage 29 and is discharged tangentially into the chamber within the member 20 and outside the pipe 23.

The gas discharged from the passage 29 is flowing at high velocity and when discharged tangentially into the chamber within the member 20, the gas flows around and around within the hood member 20 at high speed so that entrained liquid carried by the gas is thrown radially outwardly against the member 20 and then drains down into the saturator bath. The entrained liquid is thereby removed from the gas. The gas within the member 20 gradually flows downwardly and loses velocity, and then reverses its direction and flows upwardly into the pipe 23 and thence from the saturator, but by the time the gas reaches the pipe 23, substantially all of the free liquid is removed therefrom.

As pointed out above, the baffles 21 on the inside of the member 20 prevent the rapidly rotating gas within the member 20 from causing the liquid within the member 20 to swirl or rotate.

As explained above, the liquid sprayed from the nozzles 45 and 52 absorbs ammonia and other constituents from the gas, while this liquid is added to the portion of the saturator bath above the header 14, thereby increasing the concentration of ammonium sulfate in this portion of the bath. In addition, as explained above, due to the action of the pump P1 in removing liquid from the lower or bath-containing portion A of the saturator, liquid continuously flows from the portion of the bath above the header 14 through the pipe 15, from which the liquor is discharged near the bottom of the bowl 10. The liquid discharged from the pipe 15 then flows upwardly towards the suction head 62 where the liquid flows to the pump P1 and is recirculated. There is, therefore, a constant circulation of liquid in the saturator over the path which includes the suction head 62, the pump P1, the spray nozzles 45, and the by-pass pipe 64, thence downwardly through the pipe 15, and upwardly through the bath-containing portion of the saturator to the suction head 62. There is, therefore, a continuous upward movement of the liquid in the bowl 10, that is in the bath-containing portion A of the saturator.

The various parts of the lower or bath-containing portion A of the saturator are so arranged that the rate of flow of liquid upwardly in the bowl 10 is substantially uniform throughout the entire area of the bowl 10. The liquid discharged from the lower end of the pipe 15 impinges on the bottom of the bowl and flows radially outwardly and thence upwardly in the region adjacent the wall of the bowl 10, that is in the radially outer portion of the bowl 10. At the same time some of the liquid flowing downwardly through the pipe 15 is diverted by the deflectors 19 through the openings 18 and thence upwardly in the region adjacent the pipe 15, that is in the radially inner portion of the bowl 10. The liquid diverted through the openings 18 by the deflectors 19 not only creates an upward flow of liquid in the central portion of the bowl 10, but also prevents liquid discharged from the lower end of the pipe 15, and flowing upwardly in the outer portion of the bowl 10, from flowing downwardly in the region adjacent the pipe 15. Accordingly, there is an upward movement of liquid in all portions of the bowl 10, and the rate of movement of liquid upwardly is substantially uniform throughout the area of the bowl 10 at that level.

The solution which is sprayed through the nozzles 45 and is thus brought into contact with the ammonia-containing gas, is in a substantially saturated condition when it leaves the nozzles. This solution is brought to a supersaturated condition by absorption of ammonia from the gas.

Accordingly, the liquid discharged from the pipe 15 into the lower portion of the bowl 10 is a supersaturated solution. The supersaturation of this liquid is released (i. e. the liquid is desupersaturated) by contact with the crystals which are in suspension in the body of liquid in the lower or bath-containing portion of the saturator, whereupon the solution is returned to the nozzles 45 and is again brought into contact with the gas so that the cycle is repeated.

The ammonium sulfate deposited on the crystals in the lower portion of the saturator causes these crystals to grow in size so they settle to the lower portion of the bowl where they are attracted by the suction heads 70.

As the rate of flow of liquid upwardly in the bowl 10 is substantially uniform throughout the entire area of the bowl 10, the crystals settling to the lower part of the bowl 10 are of substantially uniform size throughout the area of the bowl. Accordingly, a relatively large proportion of the crystals withdrawn through the suction heads 70 are of the preferred relatively large size.

Some extremely small crystals are drawn into the suction head 62 by the pump P1 and are recirculated. These are discharged from the pipe 15 into the lower portion of the bowl 10. Some of these minute crystals will be attracted to the

suction heads 72 and will be included in the slurry withdrawn from the saturator. However, the amount of these crystals is relatively small and is not objectionable.

The rate of supply of ammonia-containing gas to the saturator, and the rate of circulation of the liquid through the saturator, are correlated in such manner that the concentration of ammonium salts in the liquid in the saturator does not materially exceed the concentration at which new crystals are formed in the absence of seed crystals, and preferably does not exceed that concentration at which new crystals form in the presence of seed crystals. This condition is known to those skilled in the art as being in the metastable but not in the labile region of supersaturation. This condition limits or represses the formation of new crystals and aids in the formation of relatively large crystals.

The bulk of the liquid circulated through the saturator flows through the nozzles 45 which are of fixed size. The by-pass pipe 64 and the valve 65 provide means for varying the rate of circulation of solution in the saturator so that the size of the crystals which are produced under varying operating conditions can be accurately controlled.

It will be seen that this saturator incorporates an acid separator for removing free liquid from the gas before the gas leaves the separator. In addition, it will be seen that the arrangement of the saturator is such that the structural elements employed in the acid-separator portion of the saturator also serve as part of other portions of the saturator.

As explained above, the cylindrical member 20, which forms the outer wall of the upper or acid-separator portion C of the saturator cooperates with the wall 12 of the central or absorber portion B of the saturator to form the arcuate duct or passage 25 through which the gas passes while being subjected to the spray from the nozzles 45.

The cylindrical member 20 also forms a part of the vertical passage 28, and, in addition, forms a part of the manifold or header 42 for supplying liquid to the nozzles 45. Furthermore, the separator portion C of the saturator, and comprising the member 20 and the head 22, is in effect an inverted vessel and cooperates with the parts associated with the duct 42 and the vertical passage 28 to enclose the upper portion of the saturator, thereby making it unnecessary to provide separate means for enclosing the top of the saturator.

It will be seen also that the lower end of the cylindrical member 20 is open, but that it extends below the level of the liquid in the saturator so that the liquid seals this end of the member 20. This not only makes it unnecessary to provide a bottom or head for the acid-separator portion C of the saturator, but it also provides a structure with a minimum of places for salt crystals to accumulate, while any salt which does build up will be dissolved during the "kill" due to the contact of the member 20 with the liquid of the bath. The arrangement of the saturator is such, therefore, that it forms a compact unit which requires a minimum of expensive material for its construction.

Subject matter disclosed but not claimed herein, for instance features which relate broadly to apparatus or method, are the subject matter of one or more of the following co-pending applications, Serial No. 338,367, filed February 24, 1953. Serial No. 338,368 filed February 24, 1953 and Serial No. 323,615 field December 2, 1952.

Although I have herein illustrated and described only one form of improved method and apparatus for removing ammonia and other constituents from coke oven gas, it is to be understood that various changes in the method or apparatus may be made without departing from the spirit and scope of my invention as set forth in the appended claims.

Having thus described my invention, what I claim is:

1. A closed vessel for separating a component of a gaseous mixture by adsorption in a liquid which forms crystals as a result of such adsorption comprising; a chamber adapted to contain a body of liquid in the lower portion and comprising a downwardly sloping bottom, means cooperating with said chamber to define arcuate elongated horizontal gas impermeable passageways for gas which are bounded at the bottom by said body of liquid, means for introducing and removing gas from said passageways, means for spraying liquid downwardly over the entire lengths of said passageways, a crystallizing chamber disposed below and adjacent the liquid-containing chamber, a conduit extending downwardly from the lowermost portion of the bottom of the liquid-containing chamber into the crystallizing chamber, the bottom of said conduit being in spaced relation to the bottom of the crystallizing chamber, means for deflecting liquid flowing downwardly in said conduit upwardly adjacent the outer periphery of said conduit, means for withdrawing liquid from the top portion of said crystallizing chamber and pumping it to said means for spraying liquid, and means for withdrawing crystals from the bottom portion of said crystallizing chamber.

2. A closed vessel for separating a component of a gaseous mixture by adsorption in a liquid comprising; an upper chamber comprising a first vertical cylindrical member, said member being open at the bottom, a lower chamber having a downwardly sloping bottom and comprising a second vertical cylindrical member of greater diameter than said first cylindrical member positioned in spaced overlapping relation thereto so as to define an annulus therewith, an arcuate closure member for the top of said annulus, an overflow conduit in said lower chamber with its top spaced above the bottom of said first cylindrical member, a gas inlet to said annulus, a gas outlet from said annulus positioned at the top of said annulus diametrically opposite to said gas inlet and communicating with the interior of said first vertical cylindrical member, a plurality of downwardly directed liquid spray nozzles positioned in the upper portion of said annulus, means for removing liquid from the bottom of said lower chamber, and means for supplying liquid to said spray nozzles.

3. A closed vessel for separating a component of a gaseous mixture by adsorption in a liquid comprising; an upper chamber comprising a first vertical cylindrical member, said member being open at the bottom, a lower chamber having a downwardly sloping bottom and comprising a second vertical cylindrical member of greater diameter than said first cylindrical member positioned in spaced overlapping relation thereto so as to define an annulus therewith, an arcuate closure member for the top of said annulus, an overflow conduit in said lower chamber with its top spaced above the bottom of said first cy-

lindrical member, a gas inlet to said anulus, a gas passageway from the top of said annulus to the interior of said first cylindrical member positioned diametrically opposite to said gas inlet, a plurality of downwardly directed liquid spray nozzles positioned in the upper portion of said annulus, means for removing entrained liquid from the gaseous mixture positioned in the upper portion of said upper chamber, means for removing liquid from the bottom of said lower chamber, and means for supplying liquid to said spray nozzles.

4. A closed vessel for separating a component of a gaseous mixture by adsorption in a liquid which forms crystals as a result of such adsorption comprising; an upper chamber comprising a first vertical cylindrical member, said member being open at the bottom, a lower liquid-containing chamber having a downwardly sloping bottom and comprising a vertical cylindrical member of greater diameter than said first cylindrical member positioned in spaced overlapping relation thereto so as to define an annulus therewith, an arcuate closure member for the top of said annulus, an overflow conduit in said lower chamber with its top spaced above the bottom of said first cylindrical member, a gas inlet to said annulus, a gas outlet from said annulus positioned at the top of said annulus diametrically opposite to said gas inlet and communicating with the interior of said first vertical cylindrical member, a plurality of downwardly directed liquid spray nozzles positioned in the upper portion of said annulus, a crystallizing chamber disposed below and adjacent the liquid-containing chamber, a conduit extending downwardly from the lowermost portion of the bottom of said liquid-containing chamber into the crystallizing chamber, the bottom of said conduit being in spaced relation to the bottom of the crystallizing chamber, means for withdrawing liquid from the top portion of said crystallizing chamber and pumping it to the spray nozzles, and means for withdrawing crystals from the bottom portion of said crystallizing chamber.

5. A closed vessel for separating a component of a gaseous mixture by adsorption in a liquid comprising; an upper chamber comprising a first vertical cylindrical member, said member being open at the bottom, a lower chamber having a downwardly sloping bottom and comprising a second vertical cylindrical member of greater diameter than said first cylindrical member positioned in spaced overlapping relation thereto so as to define an annulus therewith, a flat arcuate closure plate extending around the top of said annulus, an overflow conduit in said lower chamber with its top spaced above the bottom of said first cylindrical member, a gas inlet to said annulus, a gas outlet from said annulus positioned at the top of said annulus diametrically opposite to said gas inlet and communicating with the interior of said first cylindrical member, an arcuate liquid manifold disposed above and employing as its bottom boundary said flat arcuate closure plate, a plurality of downwardly directed liquid spray nozzles in communication with said liquid manifold and positioned in the upper portion of said annulus, means for removing liquid from the bottom of said lower chamber, and means for supplying liquid to said liquid manifold.

6. A closed vessel for separating a component of a gaseous mixture by adsorption in a liquid comprising; an upper chamber comprising a first vertical cylindrical member, said member being open at the bottom, a lower chamber having a downwardly sloping bottom and comprising a second vertical cylindrical member of greater diameter than said first cylindrical member positioned in spaced overlapping relation thereto so as to define an annulus therewith, a closure member for the top of said annulus, an overflow conduit in said lower chamber with its top spaced above the bottom of said first cylindrical member, a third vertical open ended cylindrical member of lesser diameter than said first cylindrical member positioned in the upper chamber with its bottom at a level above the top of said overflow conduit, a gas inlet to said annulus, a gas passageway from said annulus positioned at the top of said annulus diametrically opposite to said gas inlet and shaped to deliver gas tangentially to the interior of said first cylindrical member, a plurality of downwardly directed spray nozzles positioned in the upper portion of said annulus, means for removing liquid from the bottom of said lower chamber, and means for supplying liquid to said spray nozzles.

JOSEPH VAN ACKEREN.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
853,517	Osborne	May 14, 1907
1,264,263	Brassert	Apr. 30, 1918
1,895,652	Fisher	Jan. 31, 1932
1,936,308	Mueller	Nov. 21, 1933
2,375,922	Jeremiassen	May 15, 1945
2,424,205	Otto	July 15, 1947
2,424,206	Otto	July 15, 1947
2,439,547	Otto	Apr. 13, 1948
2,482,683	Tiddy	Sept. 20, 1949

Note

Page 397 (sheets 1, 2) are on card 10

p. 401 (1)

Ammoniumsulfat. Wirtschaftlicher als die älteren Dest.-Kessel arbeiten die Säulen, weil in ihnen das Gaswasser nach dem Gegenstromprinzip dem Dampf entgegenzieht. Man kann die Dest. in einer Kolonne zu Ende führen, oder man treibt das NH_3 der fixen Salze, die erst unter Zusatz von Kalkmilch zerfallen, in einem Nebenapp. aus. Säulen gewöhnlich beträchtlich hoch; mehrfache Konstruktionen. In den Hohlraum der Säule eingesetzte Querwände haben für den Dampfdurchtritt mit Glocken überdeckte Stützen und Ueberlaufrohre für die Fl. (vgl. S. 269). Das durch die Abhitze der Dest. vorgewärmte Gaswasser tritt stets oben ein, die Kalkmilch etwa im unteren Drittel. Die Ablaugen laufen fortwährend ab. Aus dem beim Dest. entweichenden Dampfgasgemisch (NH_3, W.-Dampf, CO_2, H_2S) bindet man in einem geschlossenen App. das NH_3 an H_2SO_4, während die andern Gase durch ein Abzugsrohr weitergehen. Die starke Erwärmung beim Neutralisieren verhindert das Kondensieren von W.-Dampf.

Der App. heißt der Sättiger (Abb. 19); unten konisch zulaufender Zylinder, Wandungen aus Blei, auch aus säurefesten Steinen in einem Eisengefäß. Unter ständigem Zuführen von H_2SO_4 (60° Be) fällt $(NH_4)_2SO_4$ aus und setzt sich in dem konischen Teil ab[1]). Von ihm gelangt es durch einen mit Dampf betriebenen Ejektor *E* auf eine Ablaufvorrichtung, von der aus die Mutterlauge wieder in den Sättiger zurückfließt, während ihre Reste aus dem noch stark feuchten Salz abgeschleudert werden. Trocknen in der Wärme liefert das $(NH_4)_2SO_4$ des Handels mit etwa 25% NH_3.

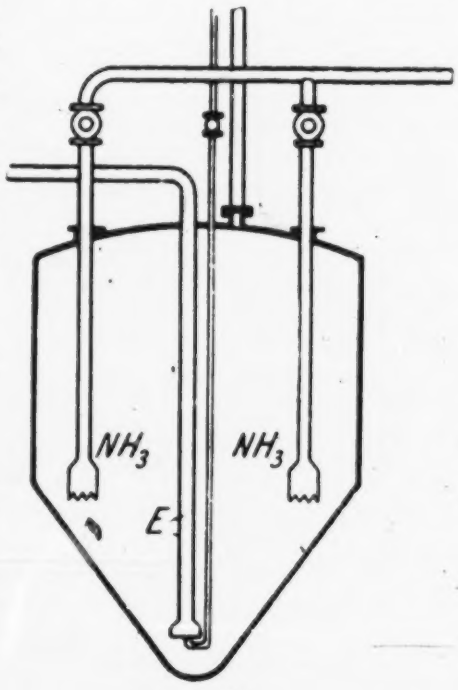


Abb. 19.

Der Kolonne darf nicht mehr NH_3-W. zugeführt werden, als der Sättiger aus ihm NH_3 aufnehmen kann, damit kein NH_3 in die großen Mengen der Abwässer gelangt. Auch soll nicht mehr W.-Dampf als nötig durch die Säule gehen, damit nicht im Sättiger die Rk.-Temp. sinkt. Die Rohrleitungen müssen über 70° haben, damit nicht durch B. von $(NH_4)_2CO_3$, das erst bei 68° dissoziiert, Verstopfungen eintreten.

Ammoniak erhält man bei der vorigen Arbeitsweise, wenn man die Kalkmilch auch in den obersten Teil der Säule führt. So können CO_2 und H_2S an CaO gebunden und in den Ablaugen mit fortgeführt werden. Man kann auch die Gase wie bei Darst. von $(NH_4)_2SO_4$ entwickeln und sie in einem besonderen Wäscher durch Kalkmilch schicken. W.-Dampf wird in einem Röhrenkühler (S. 255), Empyreuma und Pyridin werden durch Holzkohle entfernt. Das noch mit Natronlauge gewaschene NH_3 wird in W. geleitet und kommt gewöhnlich mit 25% NH_3 als Salmiakgeist von 0,910 spez. G. in den Handel; enthält Spuren von Verunreinigungen, außer den Bestandteilen des gewöhnlichen W. meist noch Empyreuma. Zur Gewinnung von chem. reinem wss. NH_3 werden die Gase weiter mit Paraffinöl gewaschen und über trockenen Aetzkalk geleitet; zuletzt wird auch nicht in eisernen, sondern in kupfernen Gefäßen gearbeitet. (Synthetische Darst. von NH_3 s. S. 775.)

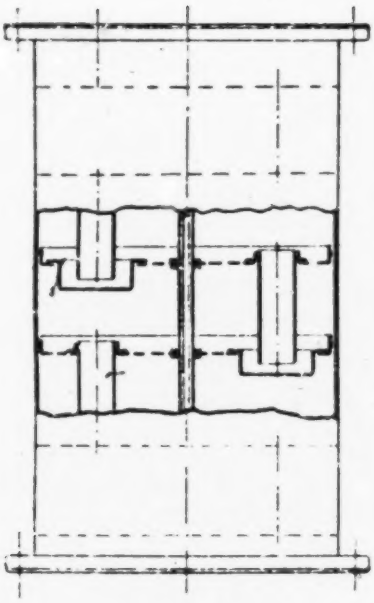


Abb. 14.

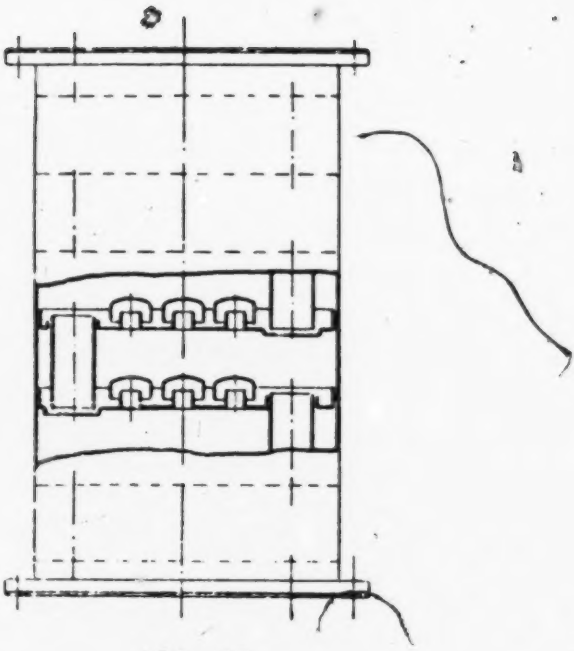


Abb. 15.

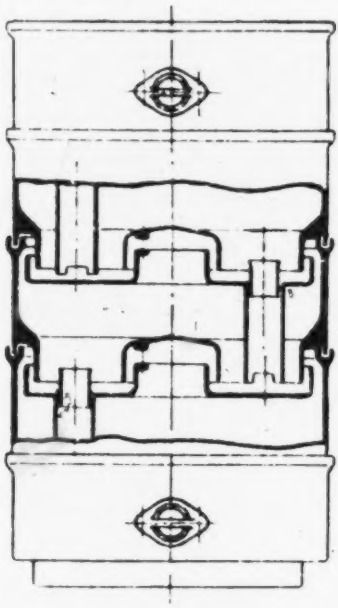


Abb. 16.

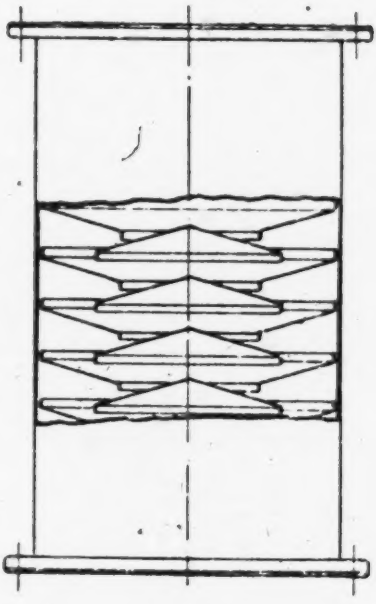


Abb. 17.

Plaintiff's Exhibit 40.

TRANSLATION

"Hütte"

Taschenbuch für den praktischen Chemiker
(Handbook for the practical chemist)

1927, 2nd edition, pp. 818, 819

* * * * * * * * * *

p. 818

2. The processing of gas liquor to ammonium sulfate and ammonia.

Ordinary gas liquor contain 1-3% NH_3, seldom more, in the form of salts, that from gas works far remote from the processing plant also 20-25% NH_3 from considerations of transportation charges. Of the NH_3 salts some (such as the carbonate, sulfate, cyanide) undergo decomposition already by mere boiling, the others (such as the sulfate thiosulfate, sulfocyanide, and ferrocyanide) with the cooperation of CaO. The crude gas liquor stocked in covered containers is clarified and distilled in column apparatuses.

Ammonium sulfate. Operating more economically than the older drum still are the columns as in these the gas liquor flows counter to steam according to the counter current principle. The distillation can be carried to completion in a column, or the NH_3 of the fixed salts, which undergo decomposition only with addition of milk-of-lime, in a subsidiary apparatus. Columns usually have a considerable height; several constructions. Transverse partitions mounted inside of the column cavity are fitted with nipples serving as a passageway for the steam and overflow pipes for the liquor (see page 269). The gas liquor, preheated by the waste heat from the distillation, is always admitted at the top, the milk-of-lime about in the lower third. The waste liquors are drained off continuously. Of the steam-gas mixture escaping during the distillation

Plaintiff's Exhibit 40.

(NH_3, steam CO_2, H_2S) the NH_3 is caused to combine with H_2SO_4 in a closed apparatus, whereas the other gases are abducted through a vent pipe. The large amount of heat liberated during the neutralization prevents the steam from condensing.

p. 819.

The apparatus is called "saturator" (fig. 19); cylinder merging into the lower conical bottom, walls lined with lead, or acid-resisting blocks in an iron tank. Upon the continuous addition of H_2SO_4 (60° Be.) $(NH_4)_2\ SO_4$ precipitates and is collected in the conical part. 1) Thence it is delivered by a steam-operated ejector E into a decanter from which the mother liquor is run off again into the saturator whilst its residual traces are separated by centrifugal action from the still very moist salt. Desiccation in the heat affords the commercial $(NH_4)_2\ SO_4$ with about 25% NH_3.

No more NH_3—water shall be fed into the column than the saturator can extract NH_3 therefrom in order that no NH_3 find its way into the large volume of waste liquors. Nor should more steam be passed through the column than is necessary in order that the reaction temperature does not drop in the saturator. The tubular conduits must be held at above 70°C. in order that there be no clogging-up due to the formation of $(NH_4)_2\ CO_3$ which dissociates only at 68°C.

Footnote reference:

1. E. Terres & W. Schmidt, Gas-und Wasserfach 1927, vol. 70, p. 725.

Plaintiff's Exhibit 2-A to Byrns' Deposition.

WOODWARD IRON COMPANY

WOODWARD, ALA.

From: L. A. Pierce

To: P. E. White

Subject: Inspection trip to Sparrows Point, Lackawanna and Pittsburgh.

Copy to: H. A. Byrns, C. W. Phillips and file.

The trip had two purposes: to inspect new developments in ammonium sulphate equipment and to learn of corrosion problems and correction in the Benzol plant pure still. This report will be divided as to these subjects rather than to the plants and operators visited.

The new Otto saturator at Lackawanna is obviously the most radical advancement in ammonia recovery in several years. This installation differs from proposed similar saturators in that no provision for a cracker pipe is made in future jobs and no acid separator is to be provided. Other changes will be made that will considerably reduce the cost. (The Lackawanna saturator is a refinement of the Northampton saturator.) To simplify the descriptions, only the proposed saturators will be described.

There is no usual scrubbing of the gas in that no tall packed column or cracker pipe is used. The saturator actually will hold no bath. Ammonia removal is accomplished by five sprays that distribute the mother liquor over the gas stream in such a way as to approach complete removal and involve no entrainment of particles of liquor. A three inch spray is provided in the inlet pipe discharging into the saturator. Three 3 inch sprays are located around the shell about two-thirds of the way up the saturator. A six inch spray is located at the top, just below the outlet pipe. At one time a spray was to be installed

in the acid separator for further removal, but the entire apparatus has now been eliminated, I understand.

All material in the saturator is monel, except at the point where acid is introduced. Monel cannot withstand conditions attending strong acid dilution, so lead is used at these points. Great claims are made for the corrosion and erosion resistance of monel.

At Lackawanna, considerable difficulty was first experienced in making a salt of sufficient crystal size that it could be dried. Experiments still being conducted have revised operating practice to the point that a satisfactory sulphate is produced. Screen analyses of salt indicates uniform crystal size, but I am inclined to be skeptical about all the analyses reported. Screen analyses are most difficult to make as the salt has a strong tendency to agglomerate.

Examination of the salt proves it to be a good salt, but not as good as screen analyses indicates. Crystals are not large but tend to be uniform and of a satisfactory size for fertilizer blending.

There are several criticisms of the saturator—some of which are apparently unfounded. It has been said that the Otto Saturator will not recover pyridine. I understand pyridine has been recovered, but that other difficulties have prevented the continued operation of their pyridine plant at Lackawanna. The monel metal used is supposed to be 60 times as subject to acid corrosion as 316 stainless according to International nickel tests conducted in 1941. More recent tests under actual operating conditions have shown the monel to be superior to 316, I understand. (I have not seen the figures on these latter tests, but have been told about them.) A fifteen month operating period at Northampton plant showed absolutely no signs of corrosion, I understand.

The elimination of the acid separator might not prove entirely satisfactory. On the basis of extreme carryover

Plaintiff's Exhibit 32.

(See Opposite ☛)

Note

Page 399 is on card 12.

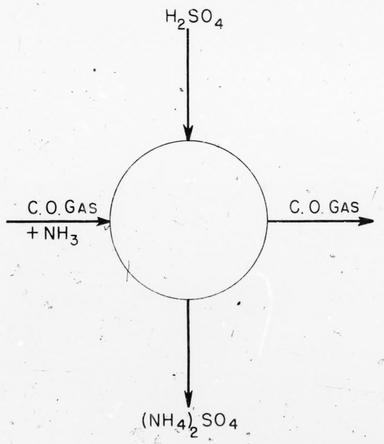
Plaintiff's Exhibit 40.

(See Opposite ☛)

SATURATED AND SUPERSATURATED

SOLUTIONS OF AMMONIUM SULFATE CONTAINING SULFURIC ACID USED FOR ABSORPTION OF NH_3

GENERAL PROCESS



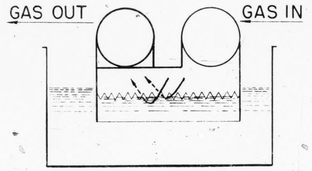
UNSATURATED SOLUTIONS OF AMMONIUM SULFATE CONTAINING SULFURIC ACID USED FOR ABSORPTION OF NH_3

MADE BY

BRUNCK 773,784 (1904)

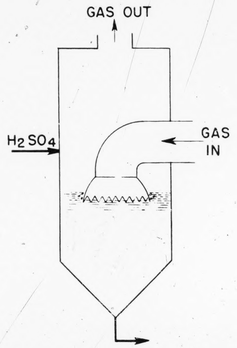
EARLY CRACKER-PIPE DESIGN. USES CONCENTRATED ACID ON HOT RAW GAS.

USES OPEN VATS TO AVOID STOPPAGES IN PATH OF GAS.



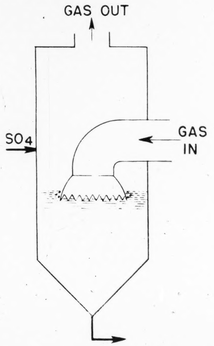
BECKER 1,747,616 (1930)

CONVENTIONAL CRACKER-PIPE DESIGN.



ECKER 1,747,616 (1930)

IVENTIONAL CRACKER-PIPE IGN.

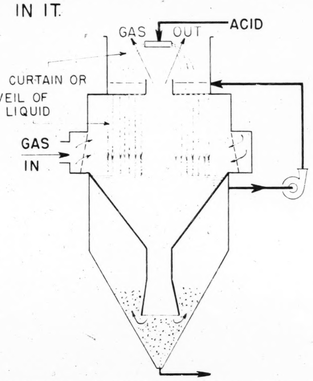


JEREMIASSEN 2,375,922 (1945)

USES SATURATED SOLUTION WITHOUT CRYSTALS.

USES CURTAIN OR VEIL OF LIQUOR FOR GAS-LIQUID CONTACT. THIS IS NOT A SPRAY.

USES DISTRIBUTING PLATE WITH HOLES IN IT.

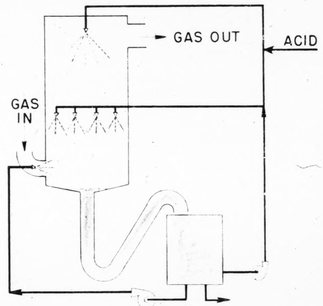


OTTO 2,599,067 (1952)

SPRAYS SATURATED SOLUTION WITH ENTRAINED CRYSTALS.

CRYSTALS GROW IN THE LIQUOR DROPS FALLING THROUGH GAS.

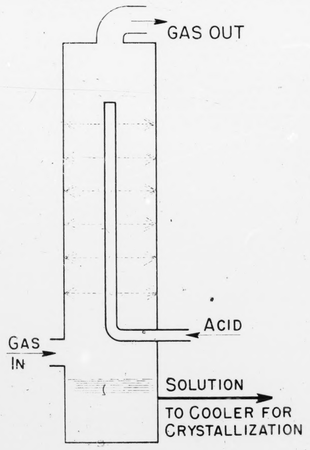
THE ONLY DESIGN EXCEPT CRACKER-PIPE DESIGNS THAT ACCOMPLISHES SIMULTANEOUSLY TWO OPERATIONS; ABSORPTION AND CRYSTALLIZATION.



NEUMEYER G.P. 24,511 (1883) SEE ALSO
WELLSTEIN G.P. 28,762 (1884)

SOLUTION ONLY IN TOWER.

CRYSTALLIZATION BY LATER COOLING.

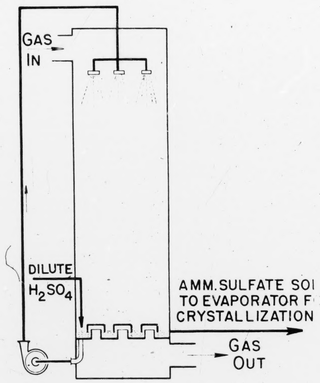


SEE ALSO BRUNCK 824,092 (1906)

DOHERTY 997,908 (1911)

OPEN TOWER. SPRAYS FROM PERFORATED PIPE CAPS.

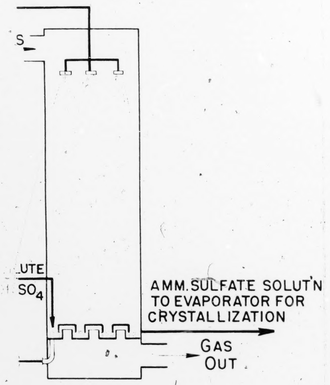
DILUTE H_2SO_4 USED → AMM. SU SOLUTION. SULFATE CRYSTALLIZ IN SEPARATE EVAPORATOR.



HERTY 997,908 (1911)

N TOWER. SPRAYS FROM FORATED PIPE CAPS.

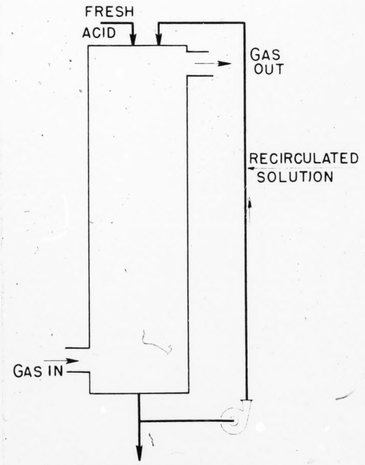
TE H_2SO_4 USED → AMM. SULF. JTION. SULFATE CRYSTALLIZED EPARATE EVAPORATOR.



BECKER 1,307,534 (1919)

HOT WASH TOWER (80°C.)

NO PRECIPITATION OF AMMONIUM SULFATE IN TOWER

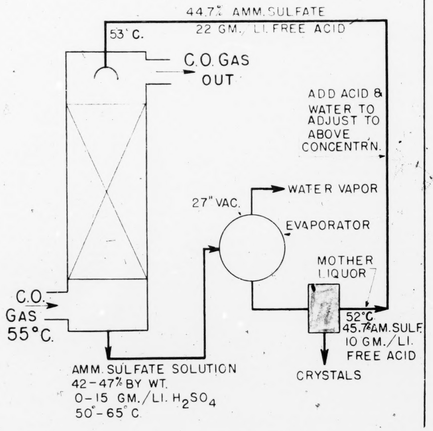


TIDDY 2,368,901 (1945 FILED 1940)

PACKED TOWER

AVOIDS SATURATION OF SOLUTION IN TOWER.

REQUIRES EXTRA STEP OF EVAPORATION PROCESS AT DIFFERENT PRESSURE (VAC.) BEFORE ANY CRYSTALS ARE MADE.



C. BOSCH & H. KELLER.

PROCESS OF ABSORBING OXIDS OF NITROGEN.

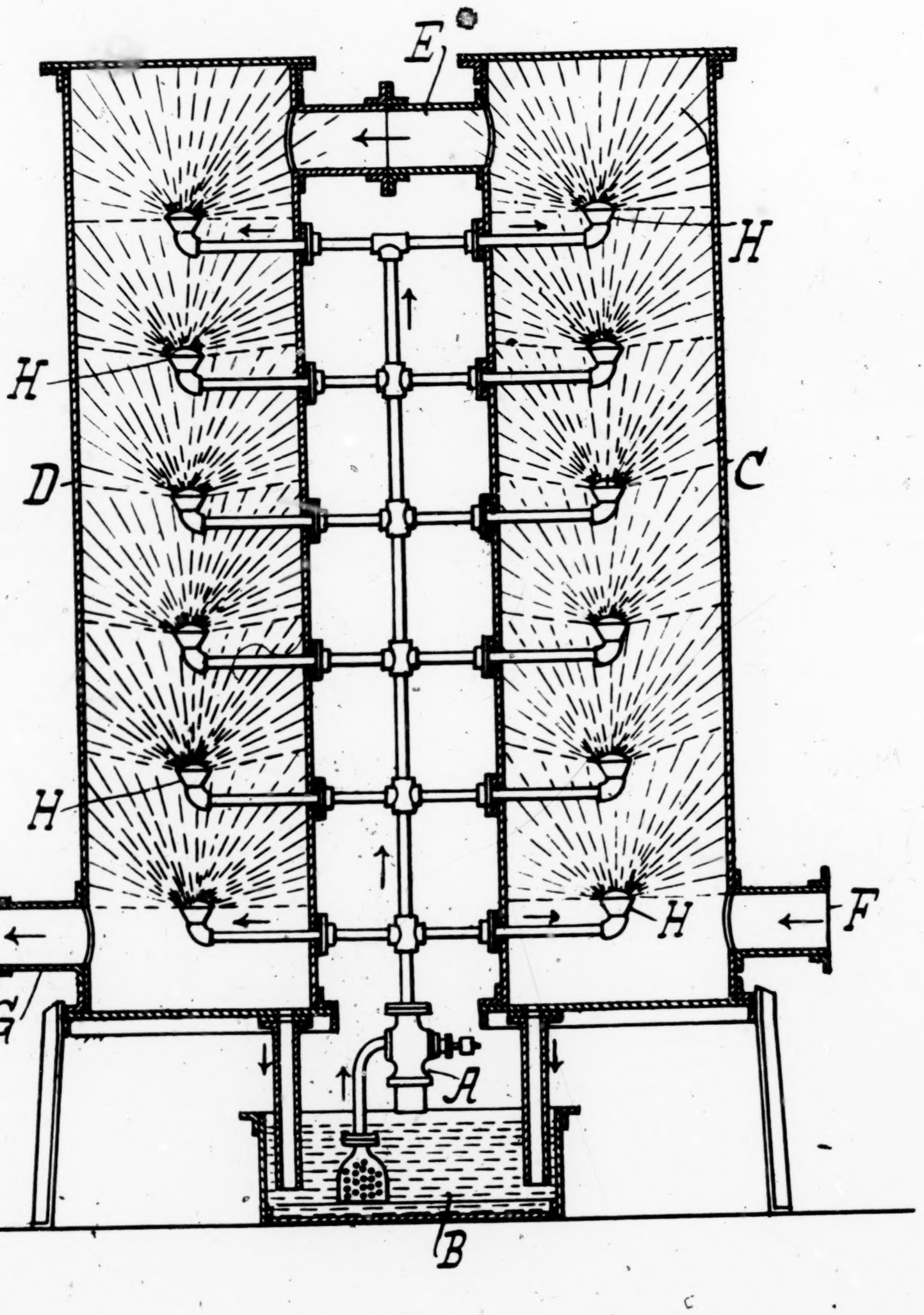
APPLICATION FILED JUNE 22, 1909.

1,029,528.

Patented June 11, 1912.

2 SHEETS—SHEET 1.

Fig. 1.



Witnesses:
William Miller
Christian W. Almstaedt

Inventors
Carl Bosch
Hans Keller
By their Attorney
W. C. Hauff

C. BOSCH & H. KELLER.

PROCESS OF ABSORBING OXIDS OF NITROGEN.

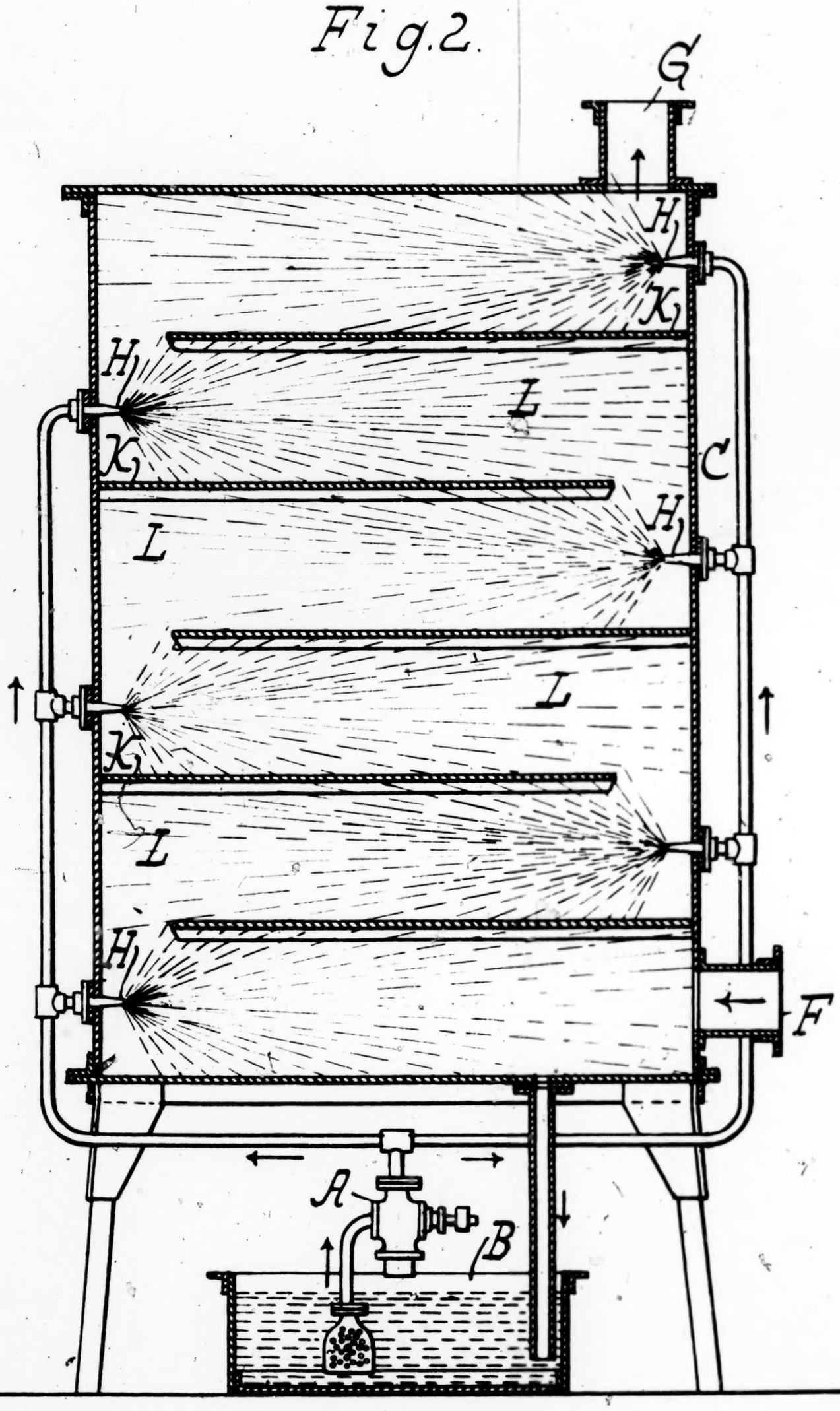
APPLICATION FILED JUNE 22, 1909.

1,029,528.

Patented June 11, 1912.

2 SHEETS—SHEET 2.

Fig. 2.



Witnesses:
William Miller
Christian H. Ahnstaedt

Inventors
Carl Bosch
Hans Keller
By their Attorney
W. C. Hanff

J. BECKER.

AMMONIA, TAR, AND BENZOL RECOVERY PROCESS.

APPLICATION FILED AUG. 12, 1918.

1,307,534.

Patented June 24, 1919.

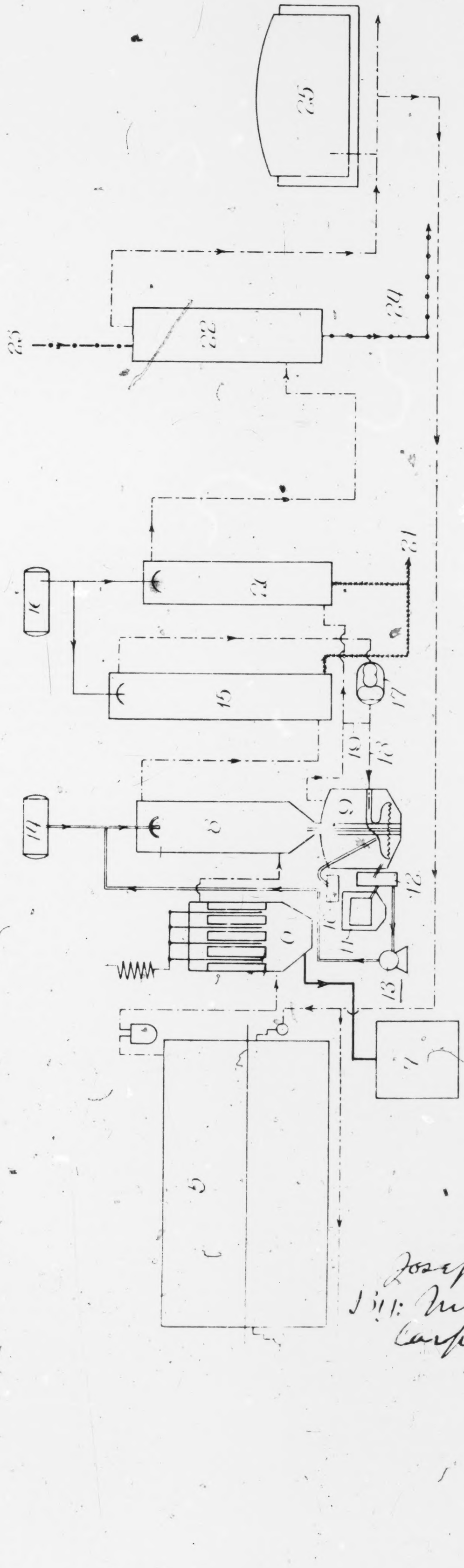


Fig. 1.

Witnesses:

Joseph Becker,

by Munday, Clarke & Carpenter, his Attorneys.

April 16, 1935. W. TIDDY 1,997,757

SATURATOR FOR THE PRODUCTION OF AMMONIUM SALTS AND PROCESS FOR MAKING SUCH SALTS

Filed Aug. 15, 1931

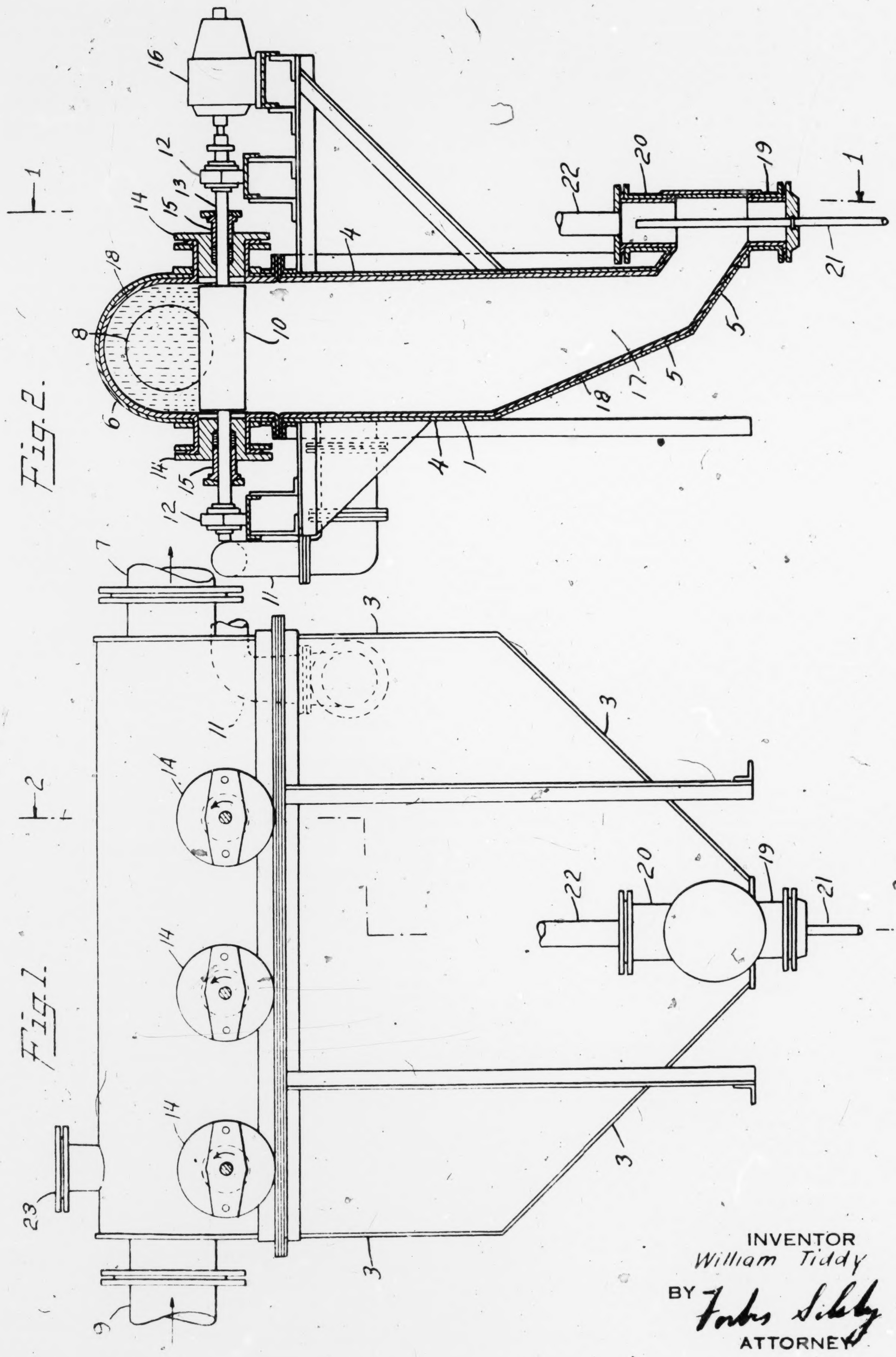


Fig. 2.

Fig. 1.

INVENTOR
William Tiddy
BY Forbes Sibley
ATTORNEY

from the saturator at Northampton, two acid separators were provided at Lackawanna. Operation without a cracker pipe at the latter installation showed such slight carryover that drain lines from the separators will not stay open. It is possible that by using two separators, gas velocity was reduced below efficient separating rates. A cyclone separator should carry 4 to 6 inches differential for optimum operation. The differential at Lackawanna was 1.5 inches. However, carryover was obviously small or even with inefficient operation enough bath would have returned through the drain lines to prevent them plugging with tar as claimed. In any event, at this point the advisability of eliminating the acid separator is dubious until actual tests prove that carryover is negligible and harmless.

It has been stated that the Otto type saturator can present operating problems in preventing the bath from going alkaline or "blue". The volumes of bath is so slight that acid feed changes or gas flow changes can result in a "blue" bath quickly if not caught by the operator. This is possible but not probable, and not a problem, according to the Lackawanna people.

Other notes on the Otto Saturator:

Bethelem men are sold on the saturator. The latest model will be installed at the Johnstown plant in the near future.

Jones and Laughlin are considering an installation at their Hazelwood plant.

Lackawanna reduced operations to 30% during the recent coal strike with no apparent operating problems.

Otto's claim that the saturator does not have to be "killed" or washed out is correct. However, it has been found that a complicated line flushing procedure can be eliminated by a bath dilution daily. This kill stops the salt for about 2 hours. No increase in acid content is

practiced as both water and acid are used for the kill. They prefer the simplified routine of a daily kill. Ammonia recovery has been found to be 99.8%, I understand.

It is believed that at the present time no other constructor can offer anything to compete with the Otto saturator. Rumor has it that Koppers has on their drawing boards something still more revolutionary—but one will not be built for 6 months yet. I attempted to learn more of the proposed Koppers saturator, but design is being carried on at such a high level (Vice-Presidents) that no one felt free to discuss it. The current Koppers saturator as proposed here is stainless steel construction and is similar in design to the Otto installation at Bethlehem's Northampton plant. It has an arrangement for improved salt crystal production that is advantageous in a critical salt market.

From my limited experience with saturators, the advantages of monel over 316 stainless and vice versa is slight.

Considering the saturator picture in relation to our needs I would say normally that it would be well to wait and see what Koppers has to offer in a few months before a decision is made. However, due to the rapidly failing condition of our saturators it might not be possible to wait that long before some decision is made. The Otto saturator seems to be entirely satisfactory and superior to anything on the market at the present. Despite the rumors that Otto is not aggressively seeking business, if a new saturator is needed in the immediate future, the Otto should be recommended, I believe, if price and delivery date are approximately equal.

Plaintiff's Exhibit 4 to Byrns' Deposition.

KOPPERS COMPANY, INC.
Engineering and Construction Division
DEVIATION FROM OR ADDITIONS TO
CONTRACT AND SPECIFICATIONS

CONTRACT WOODWARD IRON COMPANY—M.O. 528-48
SUBJECT AMMONIA ABSORBER ALTERATIONS
PURPOSE To improve operation

.....

CHANGE REQUESTED BY D. J. M3Farlin C.R. #....
DWGS. 302-A717, 302-A718, 302-A722, and 302A-933

DEV. NO. 1
DATE November 18, 1949
ACCT. NO. MO-528-48
CLAUSE AFFECTED
(CONT. OR SPEC.) 1-a, 2-a, 10-a
LETTER September 28, 1949

DESCRIPTION

The following alterations are to be made to the Ammonia Absorber installation:

Replace present slurry circulating pump with a Worthington pump, Type 10 CGL-1, 880 R.P.M. 2500 G.P.M. 35 ft. differential head. Replace 40 H.P. motor with a 50 H.P. motor.

Provide a 24″ manhole on Ammonia Absorber shell, remove baffle at gas inlet, remove outside vent pipe, add new 6″ vent pipe inside Absorber, add water spray pipes to keep vent open, remove plates on 16″ slurry suction nozzle inside of Absorber down to centerline of nozzle, add notch in hood over tar skimmer outlet pipe, replace 20 splash plate nozzles with 20, 3″—13A Spraco nozzles, change the two mother liquor splash plate sprays to one 2″ 11A Spraco Nozzle, remove all baffle plates in gas compartment of Ammonia Absorber, change one water spray in gas outlet pipe to two.

Plaintiff's Exhibit 1 to Phillip's Deposition.

KOPPERS COMPANY, INC.
General Offices
Pittsburgh - 19 - Pa.

October 17, 1949

Mr. C. W. Phillips, Chief Engineer
Woodward Iron Company
Woodward, Alabama

RE: M. O. 528-48
COMBINED AMMONIA ABSORBER AND ACID SEPARATOR

Dear Sir:

After placing the Ammonia Absorber in operation at Neville Island, we found it desirable to make some changes, which we intend to incorporate in your Ammonia Absorber also.

As this equipment has already been delivered, it will be necessary for you to make these alterations in the field:

1. A new slurry circulating pump and motor having a higher differential head is being bought to replace the pump and motor originally furnished. New pieces of stainless steel pipe will be furnished for altering present pipe to suit new pump.

2. New spray nozzles will be furnished to replace present spray nozzles for slurry circulating and mother liquor.

3. Ammonia Absorber Alterations

   Add manhole to Absorber Shell.
   Remove baffle at the gas inlet nozzle.
   Remove all baffles in gas compartment.
   Remove outside vent pipe.
   Add new vent pipe inside Absorber
   Add water spray pipes to keep inside vent pipe open.
   Remove plates on 16" slurry suction nozzle inside of Absorber down to centerline of nozzle.
   Remove two mother liquor spray pipes and replace with one mother liquor spray pipe located in cen-

ter of gas outlet passage and add two water sprays, one on each side of mother liquor spray.
Cut notch in hood over Tar Skimmer outlet pipe.
Add water spray in gas inlet pipe.

Drawings showing details of all alterations are being prepared and will be forwarded shortly. As these changes will affect the Slurry Circulating pump foundation, no further work should be done until the new drawings are received.

Yours very truly,

KOPPERS COMPANY, INC.
Engineering & Construction Division
C. E. MIELKE
C. E. Mielke
Contract Engineer

Plaintiff's Exhibit 2-A to Phillips' Deposition.

August 12, 1948.

Koppers Company, Inc.,
Engineering & Construction Division,
Pittsburgh 19, Penna.

Attention—Mr. J. W. Greene.

Gentlemen:—

With reference to your Specifications and Quotation for saturator and auxiliaries, we understand that this is an entirely new and, therefore, untried design.

We also have the impression that Koppers Company stands behind this design.

Will you please, therefore, advise what guarantee of performance you propose on this installation.

Yours very truly,

WOODWARD IRON COMPANY,
J. H. Leonard,
Purchasing Agent.

Plaintiff's Exhibit 2-B to Phillips' Deposition.

August 17, 1948

Mr. J. H. Leonard
Purchasing Agent
Woodward Iron Company
Woodward, Alabama

SUBJECT: NEW SATURATOR EQUIPMENT
PROJECT 3892

Dear Mr. Leonard:

In reply to your letter of August 12, 1948, with reference to New Saturator and Auxiliary Equipment outlined in our Specifications dated July 19, 1948, Project 3892, we wish to advise as follows:

The Seller agrees that all workmanship furnished by Seller or its contractors covering New Saturator and Auxiliary Equipment shall be first class and the quality of all materials furnished shall be in every respect suitable for the purpose for which it is to be used, and this equipment will perform in a satisfactory manner in the recovery of ammonia from the gas and the resultant production of ammonia sulphate.

Yours very truly

KOPPERS COMPANY, INC.
Engineering and Construction Division

J. W. GREENE
J. W. Greene
Sales Department

(Handwritten postscript as follows):

" 'If it does not work we will make it work'

J. W. Greene

Phoned to me by Greene from Pittsburgh 8/25/48
Byrns
8/25"

Request for Admissions.

Request for Admissions, served by plaintiff on April 16, 1955, admitted by defendants:

That there are pending in the United States Patent Office patent applications serial numbers 338,367, 338,368 and 323,615, by Joseph Van Ackeren, assignor to Koppers Company, Inc.;

That the prosecution of said applications has at all times been under the control of Koppers Company, Inc.;

That in said applications specific reference is made by applicant to certain advantages and economics inherent in the spray type ammonium sulphate saturator over certain other types of saturators for treating coke oven gas;

That invention is asserted and claimed by applicant in certain improvements in spray type saturators, and that in one of said applications a claim was presented to the Patent Office which differs from another claim of said application solely by including the additional clause, "The desupersaturated solution used as a spray is undiluted and contains fine crystals of ammonium sulphate."

* * * * *

Defendants' Exhibit 14.

(See Opposite ☛)

Defendants' Exhibit 15.

(See Opposite ☛)

C. OTTO

PRODUCTION OF AMMONIUM SULFATE

2,599,067

Patented June 3, 1952

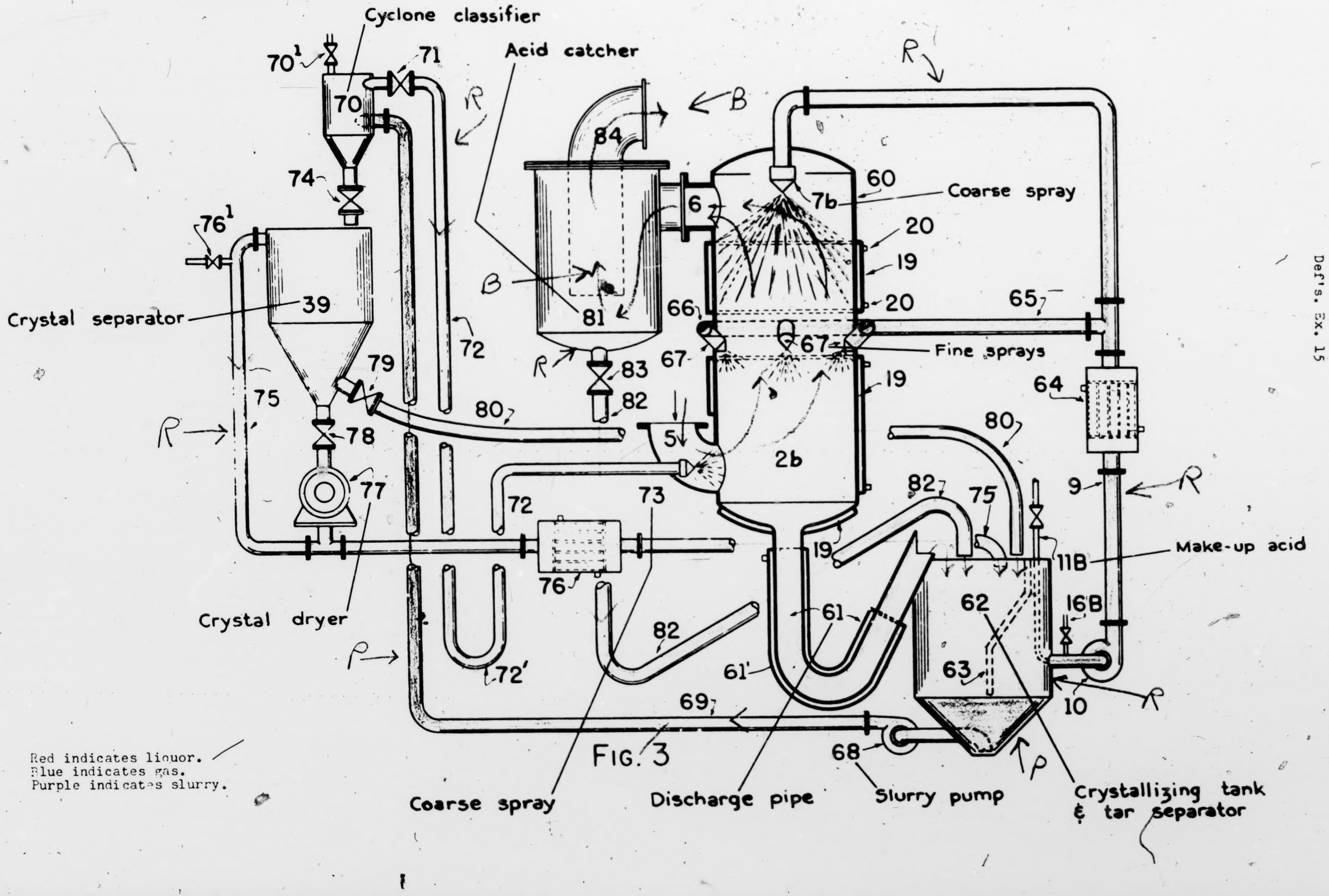


FIG. 3

Red indicates liquor.
Blue indicates gas.
Purple indicates slurry.

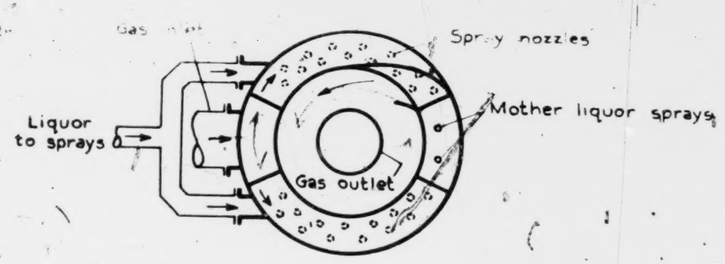
Def's. Ex. 15

Defendants' Exhibit 16.

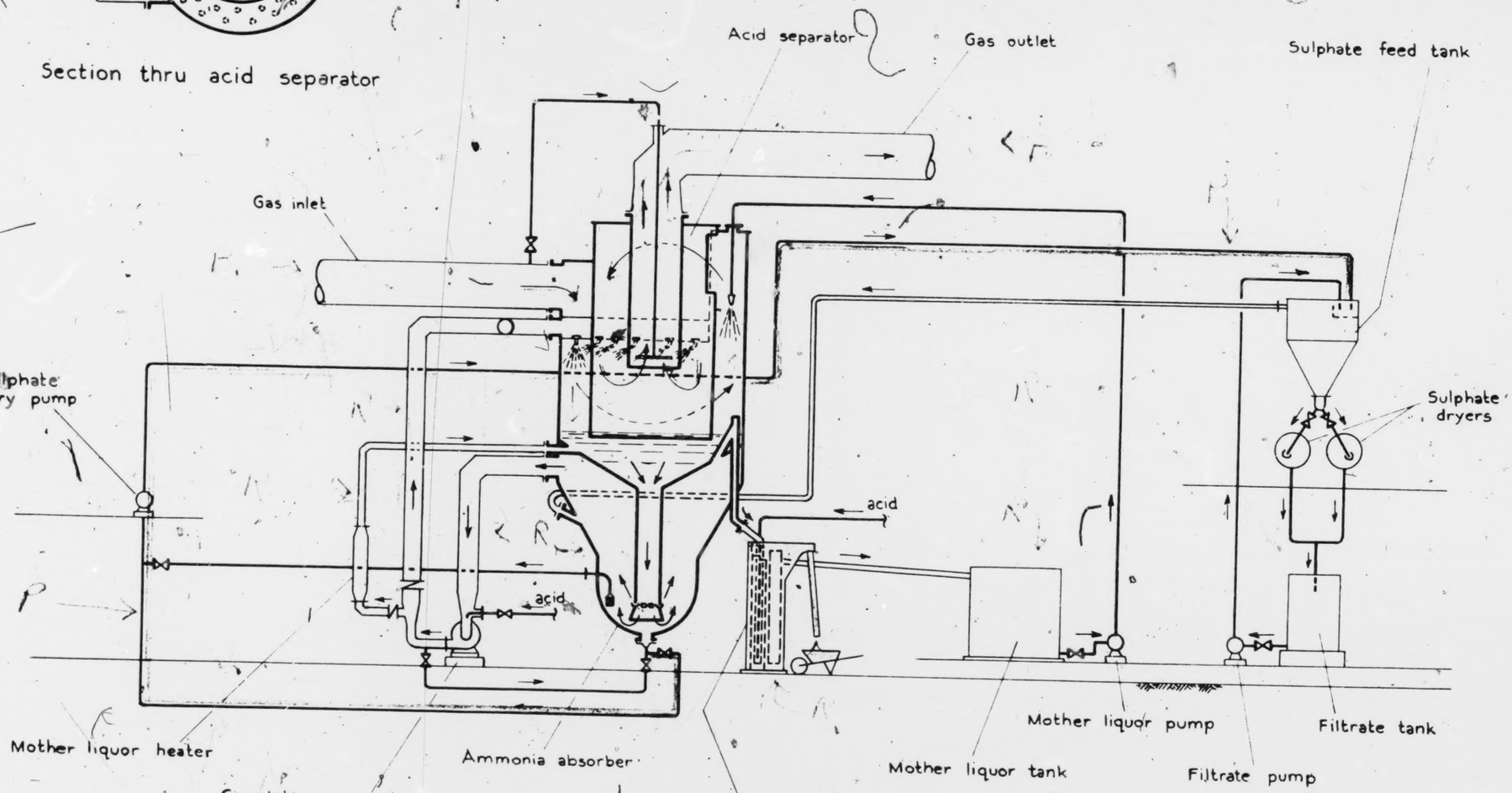
(See Opposite ☛)

AMMONIUM SULPHATE RECOVERY SYSTEM

WHEELING STL. CO. - CONTRACT # 1850



Section thru acid separator



Red indicates liquor.
Blue indicates gas.
Purple indicates slurry.

Koppers Co., Inc. drawing No. 1850-2A10
(Simplified)

Defts. Ex. 16

Defendants' Exhibit 18.

Patent No. 997,908.

(See Opposite ☞)

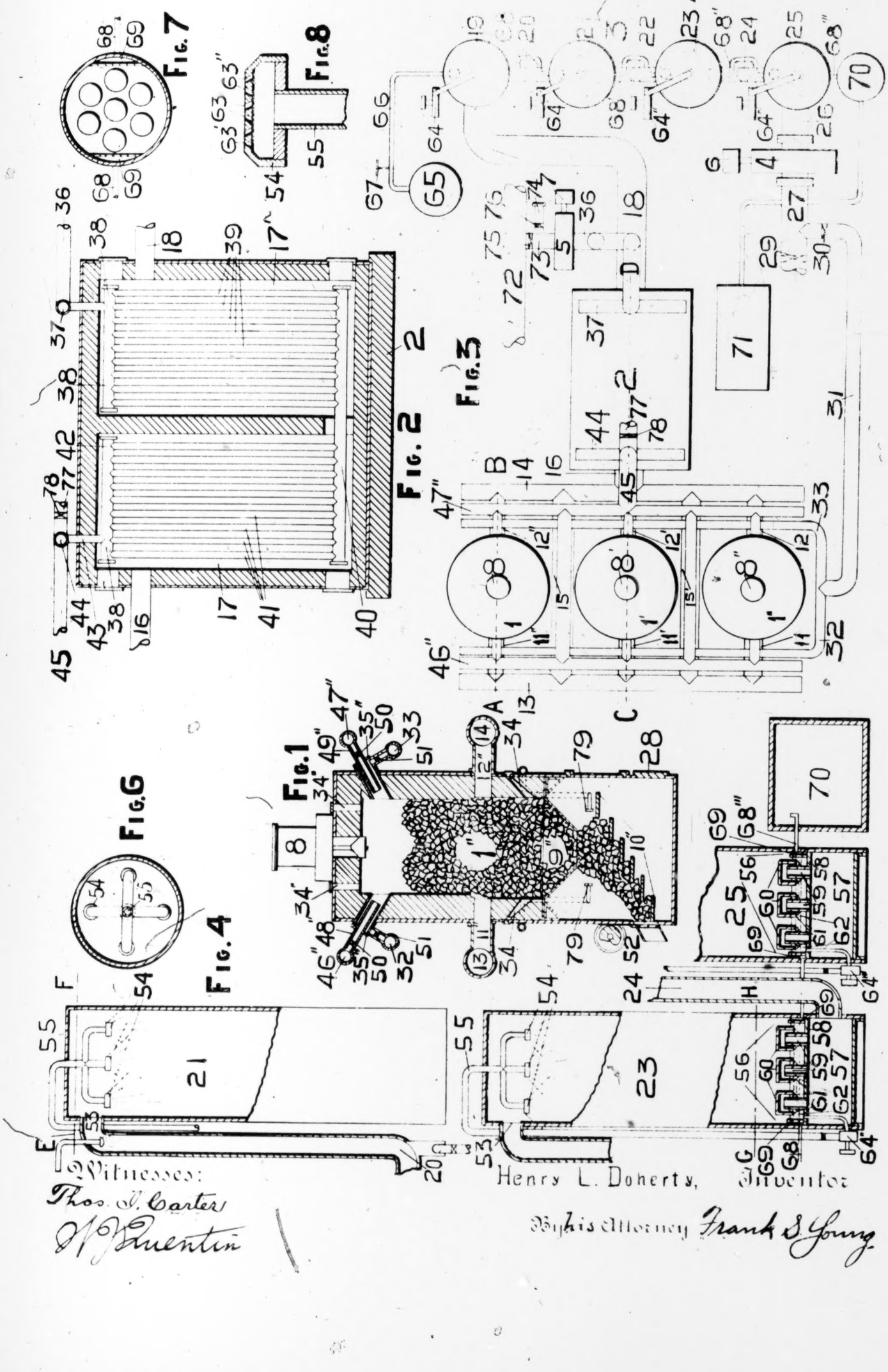
H. L. DOHERTY.

PROCESS OF OBTAINING AMMONIA FROM COAL.

APPLICATION FILED DEC. 23, 1909.

997,908.

Patented July 11, 1911.



UNITED STATES PATENT OFFICE.

HENRY L. DOHERTY, OF NEW YORK, N. Y.

PROCESS OF OBTAINING AMMONIA FROM COAL.

997,908. Specification of Letters Patent. Patented July 11, 1911.

Application filed December 23, 1909. Serial No. 534,693.

To all whom it may concern:

Be it known that I, HENRY L. DOHERTY, a citizen of the United States, residing at New York city, in the county of New York and State of New York, have invented new and useful Improvements in Processes for Recovering as Ammonia the Volatile Nitrogen of Coal, of which the following is a specification.

This invention relates to processes for recovering as ammonia the volatile nitrogen of coal and, in particular, to such processes in which the volatile nitrogen of the coal is caused to be evolved as ammonia by subjecting the coal to destructive distillation in closed chambers.

The object of my invention is the conversion of the greatest practicable proportion of the volatile nitrogen of the coal to ammonia by the destructive distillation of the coal and subsequent preservation in, and recovery from, the distillation gases. I accomplish these objects by carrying out the [illegible] in an atmosphere [illegible] favorable to the formation of ammonia, while maintaining [illegible] temperature in the distillation chamber sufficient for perfect carbonization, regulating and controlling the temperature to which the evolved gases are subjected that the latter may at no time exceed a temperature above that essential to such carbonization.

In the accompanying drawing I show a form of apparatus by means of which my process may be carried out.

Figure 1 is a vertical section through one of the carbonizing chambers, taken on a plane through the line A B of Fig. 3; Fig. 2 a similar section through the recuperator taken on a vertical plane through the line C D of Fig. 3. Fig. 3 is a diagrammatic plan of the apparatus; Fig. 4 shows a section of the upper part of tower 21 with a portion of the gas pipe connecting 19 and 21. Fig. 5 shows a part section through the last two towers 23 and 25 showing one of the sprays, one of the liquid seals, overflow, etc. Fig. 6 is a section through one of the towers on the line E F showing a plan of the spraying apparatus; Fig. 7 is a similar section on the line G H, showing a plan of the liquid sealing device; Fig. 8 is a section through one of the spray caps.

The method of operation is as follows: In starting the apparatus, coarse cinder or coke is charged into the carbonizing chambers, 1, 1', 1'', through the charging mechanism, 8, 8', 8'', until a bed has been built up in each carbonizing chamber well above its hopper bottom, which latter, in the chamber shown in section, is designated 9''. 10'' is the stepped grate of the carbonizing chamber, which, with 9'' supports the charge therein. When a sufficient quantity of cinder (or coke, as the case may be) has been charged into the carbonizing chambers, a fire is kindled on the bed and coke gradually charged through the hoppers 8 until there has been built up in each chamber a bed of ignited coke to above the gas "off-takes" 11'' and 12''. The gas "off-takes," 11, 11', 11'', connect with the pipe 13, the "off takes" 12, 12', 12'', on the opposite side of the carbonizing chambers, with the pipe 14. These pipes 13 and 14 are joined by the pipes 15 and 15', the main gas conduit, 16, receiving the gas flowing through them and conducting it to the gas chamber, 17, of the recuperator 2. The discharge conduit, 18, from the recuperator connects with the tower 19 of the absorption apparatus 3. The last absorption tower, 25, is connected with the exhauster 4 by the pipe 26. When the fire has been kindled in the carbonizing chambers, the blower 5 is started at a low speed. The doors 8a of the chamber being open, air enters below the grate 10'' and passes up through the material resting on the grate, and sustains the combustion in the carbonizing chamber. The combustion gases are drawn off through 11'' and 12'' and are conducted through the conduit 16 to recuperator 2, through the gas passages of 2 and the passage 18 to the first absorption tower, 19, thence through 19' 21, 23 and 25 and their connecting passages to the exhauster and discharge through the pipe 27. The hot gases may be cooled previous to their entry to the exhauster, by forcing air through the recuperator by means of the blower 5, allowing the hot air to waste through the pipe 77 controlled by valve 78, or by circulating a cooling liquid in the absorption towers in the manner explained below.

At first the gases discharging through 27 are principally flue gases, the combustion, at first, being the complete combustion to CO_2, owing to the thinness of the fuel bed. As the thickness of the bed of burning coke increases the character of the gases changes.

in 11, when the fuel bed has attained to the level of 11″ 12″ the combustion of the carbon has changed, in greater part, to the incomplete combustion to CO, and a fairly good producer gas is being drawn off from the chambers. The valve 29 is now partially closed and the valve 30 opened, part of the gas passing back through the pipe 31 to the gas distributing-pipes 32 and 33. By means of a torch, or a metal rod heated to incandescence, which is introduced through the poke-holes, 34″, and the corresponding poke-holes of the other carbonizing chambers, the gas is ignited as it issues from the gas burners 35″. The air for supporting the combustion at the burners 35, of the several carbonizing chambers, is supplied under pressure by the air-blower 5, the valve 78 being closed, and passes through the pipe 36 to the distributing pipe 37 of recuperator 2. From 37 it is distributed to the headers 38, of the air flues 39 of the recuperator, passed down through 39 to the lower headers 40, up through the vertical flues 41 to the headers 42, through the connections 43 to the cross-connection 44, and thence from 44 to the air distributing pipes 46 and 47 of the carbonizing chambers, through the pipe 45. From the air distributing pipes 46″ and 47″ the air passes through the tubes 48″ and 49″ of carbonizing chamber 1″ and the corresponding air tubes of the other carbonizing chambers. Valves 50″ and 51″ on the air and gas tubes, respectively, of the burners of chamber 1″ and similar valves, on the respective connections of the other gas burners permit of the regulation of the quantity of gas and air supplied to each burner. Coke is now charged into the chambers 1, 1′, 1″ until the bed of incandescent coke has reached to the level indicated on the drawings, in Fig. 4. The door 28 is now closed. The apparatus is now in condition to start on the carbonization of the coal. Coal is charged at intervals through the charging hoppers 8, 8′, 8″, while the material at the bottom of the chambers, resting on the grates 10, of the chambers is withdrawn in corresponding volume, through the discharging apparatus 52.

The raw coal is immediately upon its entrance to the chambers subjected to the heat of the flame from burners 35, which causes the destructive distillation of the volatile hydrocarbons of the coal. The products of the distillation and the gases from the burners 35 pass downward through the bed of fuel and thence leave the chambers through the "off take" 11 and 12 of the several carbonizing chambers, passing to the recuperator 2 by the pipe 13, shown, thence through the recuperator, where they are partially cooled by the air passing through the flues 39 and 41, through the conduits 14 to the first tower of the absorption apparatus 3. In passing through 3 the gases are cooled and the ammonia which they contain is absorbed by a suitable liquid, which is circulated through the towers.

The method of operating the absorption apparatus, 3, is as follows: As shown, the absorption towers 19, 21, 23 and 25 are each provided, with a gas inlet 53, a spraying apparatus having a plurality of spraying caps 54, connected to pipe 55, a liquid sealing device, 56, and a gas outlet-chamber, 57. The sealing device, 56, consists of a false bottom, 58, fastened water-tight into the absorption chamber and holding a small depth of liquid. Short vertical pipes, 59, are inserted in the false-bottom 58 of the chamber and are covered by caps, 60, which are supported on brackets, 61, attached to the pipes 59. These caps have a sufficiently large internal diameter to leave an annular space around 59 at least equal in area to the cross-section of the pipes 59. Under the influence of the suction of the exhauster-blower, 4, the gas enters at the top of tower 19, is loaded with spray from the spraying device 54, passes to the bottom of the tower, bubbles up through the liquid seal 62 into the caps 60, thence passes down through the pipes 59 into the chamber 57, thence through the pipe 20 to tower 21, and so on to the last tower.

The spraying device which I have shown and which I prefer to use in connection with my apparatus is the one shown in my Letters Patent No. 901,597, dated Oct. 20, 1908, and consists of a plurality of spraying caps, 54, supplied with liquid under pressure by the pumps shown, through pipes 55, 55′, etc., and having perforations in their faces. The perforations are arranged in coöperating couples, 63, 63′, etc., both perforations of each couple being located in the same plane and at the same inclination to the axis of the cap. The streams discharging from the two perforations of each couple are thus caused to intersect and impinge upon each other with the production of a very fine spray. The rapidly moving gas current carries this fine mist or fog to the bottom of the chamber where it is intercepted by the liquid seal 62, while the gas passes into the caps 60 and thence through the pipes 59 into the gas chamber 57. The backward circulation of the liquid from tower to tower in the direction of the circulation of the gas is thus prevented. The pumps 64, 64′, 64″, 64‴ maintain the liquid in circulation from the seals in the lower part of the towers to the sprays at the top. A tank or reservoir, 65, holds a supply of the fresh ammonia absorbing liquid, and this is supplied to the tower 19 through the pipe 66 at a rate corresponding with the rate at which the saturated liquid is withdrawn from the tower 25, by manipulating

the valve 67. Tubes, 68, 68′, etc., connect the seal chambers of adjacent towers, through overflow compartments 69. These compartments are formed, simply, by riveting partitions to the bottom and cylindrical wall of the seal chambers, the top of the partition being at about the level it is designed to carry the seal. As the fresh absorbent liquid flows into the seal chamber of the tower 19, therefore, an equal volume of the liquid in the seal chamber of 19 flows through the tube 68 into the seal chamber of the tower 21, and so on, the overflow from the last tower 25 passing to the storage tank 70 for the saturated ammonia liquor.

Although the gas in passing through the recuperator 2 is partially cooled, it still has a temperature of about 400° to 500° F. When, therefore, it comes into contact with the spray at the top of 19, an evaporation of water takes place with the formation of steam, the relative volumes of gas and liquid being such that the liquid in the seal of 19 is maintained at the boiling point. The pipe 20 conducting the gas from 19 to 21 is, in reality, a condenser, water being supplied at the top of 20 in sufficient quantity to cool the gas below 212° F. The steam carried out of 19 is thus condensed and collects in the bottom of 20. This condensed water holds a considerable proportion of ammonia in solution, since the liquid in 19 is nearly saturated when it is introduced into 19, and can, therefore, only take up a small additional quantity of ammonia. The condensation from 20 is therefore used in making up fresh absorbent liquor in tank 65. It is seen, from the above, that the first tower, 19, of the absorption apparatus is, in reality, a cooling and evaporating tower, the liquor drawn off from 19 to the storage 70, having its concentration considerably increased. This is accomplished by the otherwise wasted sensible heat of the gas and is therefore so much burden saved to the evaporating apparatus, proper, which is indicated at 71.

The absorbent liquid may be any of the ones in common use but will usually be a dilute solution of sulfuric acid, the ammonia absorbed being fixed as ammonium sulfate. When this is used the final evaporation in 71 is carried to the crystallizing point.

Usually during the combustion of the coal sufficient SO_3 will be formed to combine with all the ammonia formed. When this is the case the ammonia can be completely absorbed as sulfite without the introduction of any extraneously formed acid or ammonia-absorbing salt. In this case only water need be supplied to the first tower.

It is apparent, that my method of absorbing the ammonia insures the most intimate contact possible between the gas and the liquid, the absorption of the ammonia being rapid and complete. I thus reduce the capacity necessary in the absorbing apparatus to a minimum.

In the destructive distillation of coal for the recovery of ammonia there are two opposing considerations in regard to the temperature at which the distillation should be conducted. In the first place, since ammonia is appreciably dissociated in contact with carbon at even so low a temperature as 1100° F., it is apparent, that the temperature should be as low as other considerations will permit. On the other hand, in order to convert the volatile nitrogen of the coal into ammonia, it is absolutely necessary to heat the coal sufficiently high to break up the compounds in which the nitrogen is held in the coal. To secure the maximum possible initial formation of ammonia during the distillation, it has been found that the primary products of the distillation must be heated to about 1700° F. In practical working, therefore, it is necessary to strike a balance between these two opposing considerations and endeavor to operate under such conditions as will give the maximum quantity of ammonia recovered. I aim to so operate my carbonizing chambers that the maximum temperature to which the distillation products are exposed is about 1500° F.

While the raw coal may be exposed to the action of a flame of much higher temperature than that stated, owing to its low conductivity and the rapid carrying off of the heat absorbed, in the distilled vapors, the high temperature cannot be allowed to extend so far into the mass of carbonizing coal as to heat the distilled products much above 1500° F. without injuriously affecting the quantity of ammonia produced. Owing to the low conductivity of the coal and the coke formed from it, starting with a high temperature flame, the flame may penetrate to a considerable distance into the mass of material in the carbonizing chamber before its temperature has been reduced to 1500° by the secondary reactions set up. The distillation-products, therefore, would be exposed to an injuriously high temperature. To meet this condition, I, in the herein-revealed process, modify the temperature of the flame in the carbonizing chambers, by mixing with the air supplied to the burners sufficient stack or flue gases, or gases from the exhaust of an internal combustion engine taken from the flue 72 by means of pipe 73, to reduce the temperature of the flame to a safe point. By properly adjusting the valves 75 and 76 on the pipe 73 and air intake 74, respectively, I secure whatever mixture of flue gas and air I desire. By this addition I increase the heat capacity of the gases, per unit of gas

burned, by increasing the volume of the gases. I thus limit the flame temperature to the proper point, but, still, carry enough heat into the mass of coking coal to insure a proper destructive distillation of the hydrocarbons and nitrogeneous matter, with the formation of the maximum quantity of ammonia. Further, by thus increasing the normal volume of the gases, I correspondingly increase the velocity with which they pass through the mass of carbonizing coal in the chamber and thus diminish the time during which the distilled vapors and gases are in contact with the hot coke. To still further increase the yield of ammonia, I pass through the still incandescent coke in the bottom of the carbonizing chambers the steam and gases produced in the quenching of the coke in the lowest part of the coke bed, *i. e.*, the part resting upon the grates, 10, and below the hopper-bottom, 9, of the carbonizing chambers. The water is sprayed onto the coke on the grates from spray-caps, 79. The water is vaporized, by the hot coke, and rising up through the superincumbent layers of the unquenched coke, is partly dissociated into hydrogen, carbon dioxid, and carbon monoxid. The nascent hydrogen assists in the evolution of the final portion of the volatilizable nitrogen, as ammonia. In ordinary carbonization the coke retains, in some form, not at present definitely known, a large proportion of the total nitrogen of the raw coal, usually, about, 50%. By subjecting the hot coke, in the manner just described, to the influence of an atmosphere rich in nascent hydrogen I am able to convert into ammonia and recover a considerable part (about $\frac{1}{3}$) of this usually fixed nitrogen and thus greatly increase the quantity of ammonia that I recover.

Having described my invention, what I claim is:

1. The process for producing ammonia from coal, which consists, in subjecting said coal to direct contact with a gas flame, whereby the volatile nitrogeneous matter of said coal is distilled therefrom, passing the said distilled nitrogeneous matter in contact with incandescent coke, whereby the said nitrogeneous matter initially distilled from said coal is further dissociated with the formation of ammonia, and recovering the ammonia from the gaseous products distilled from said coal by said gas flame by contacting said gaseous products of distillation with an atomized liquid capable of absorbing ammonia, substantially as described.

2. The process for producing ammonia from coal, which consists, in subjecting said coal to direct contact with a gas flame, whereby the volatile nitrogeneous matter of said coal is distilled therefrom: passing the said distilled nitrogeneous matter in contact with incandescent coke, whereby the said nitrogeneous matter initially distilled from said coal is further dissociated with the formation of ammonia; and recovering the ammonia from the gaseous products distilled from said coal by said gas flame, by contacting said gaseous products of distillation with a liquid capable of absorbing ammonia, substantially as described.

3. The process for producing ammonia from coal, which consists, in subjecting said coal to direct contact with a gas flame, whereby, the volatile nitrogeneous matter of said coal and the volatile hydrocarbons of said coal are distilled therefrom; passing the said distilled matter in contact with incandescent coke, whereby, the said hydrocarbons and the volatile nitrogeneous matter, are further dissociated, with the formation of ammonia; and recovering the ammonia from the gaseous products of the reactions by contacting the said gaseous products with a liquid capable of absorbing ammonia, substantially as described.

4. The process for producing ammonia from coal, which consists, in subjecting said coal to direct contact with a gas flame, whereby, the volatile nitrogeneous matter of said coal and the volatile hydrocarbons of said coal are distilled therefrom; passing the said distilled matter in contact with incandescent coke; whereby, the said hydrocarbons and the volatile nitrogeneous matter are further dissociated with the formation of ammonia and other gases; withdrawing from contact with said coke the commingled gaseous products; cooling said products; and recovering the ammonia from said cooled products by subjecting the same to contact with a liquid capable of absorbing ammonia, substantially as described.

5. The process of recovering ammonia from coal, which consists, in coking said coal by direct contact with a gas flame; passing the products distilled from said coal in said coking operation in contact with coke from a previous charge of coal, whereby; the distilled nitrogeneous matter from said coal is dissociated with the formation of ammonia; quenching the said coke with water; passing the water vapor formed in said quenching operation in contact with incandescent coke, whereby, reactions are caused to take place between a portion of said water vapor and said coke and a portion of the nitrogen of said coke, with the formation of ammonia; withdrawing, from contact with said coke, the gases formed in the coking operation and the gases formed in the quenching operation in a common current; and recovering the ammonia from said gases by contacting the same with a liquid capable of absorbing ammonia, substantially as described.

6. The process of recovering ammonia

from coal, which consists, in coking said coal by direct contact with a gas flame; passing the products distilled from said coal in said coking operation in contact with coke from a previous charge of coal; whereby the distilled nitrogeneous matter from said coal is dissociated with the formation of ammonia; quenching the said coke with water; passing the water-vapor formed in said quenching operation in contact with incandescent coke, whereby reactions are caused to take place between a portion of said water vapor and said coke and a portion of the nitrogen of said coke, with the formation of ammonia; withdrawing from contact with said coke the gases formed in the coking operation, and the gases formed in the quenching operation in a common current; passing said gases through the gas flues of a recuperator, whereby, a portion of the sensible heat of said gases is transferred to a current of air passing through the air passages of said recuperator; conducting such heated air to the gas flame to sustain the combustion therein; conducting the gases after they leave said recuperator to an absorbing apparatus; and recovering the ammonia from said gases by contacting the said gases with a liquid capable of absorbing ammonia, substantially as described.

7. The process of recovering ammonia from coal, which consists, in subjecting said coal in an inclosed chamber to direct contact with a gas-flame of regulated intensity, the intensity of said flame being regulated by the introduction of a regulated proportion of combustion gases into the air current supplied to said flame; passing the products distilled from said coal by said flame in contact with coke from a previous charge of coal, whereby the said distilled products are subjected to partial dissociation with the formation of ammonia; and recovering the ammonia by contacting the so formed gases with a liquid capable of absorbing ammonia, substantially as described.

8. The process of recovering ammonia from coal, which consists, in subjecting said coal to destructive distillation by direct contact with a modified gas flame, said gas flame being modified by admixing with the air current supplied to said flame, a regulated proportion of combustion gases, whereby the temperature of said flame is reduced below the temperature at which ammonia spontaneously dissociates, and recovering from the gaseous products of such destructive distillation the ammonia formed by such distillation, substantially as described.

9. The process of recovering ammonia from coal, which consists, in subjecting said coal to distillation by direct contact with a modified gas flame, said gas flame being modified by admixing with the air current supplied to said flame, a regulated proportion of combustion gases, whereby the temperature of said flame is reduced below the temperature of dissociation of ammonia; passing the products of such distillation in contact with coke from a previous charge of coal, whereby, the products formed in the initial distillation of the coal are partially dissociated with the formation of ammonia and other gases; quenching the coke with water; passing the water vapor formed in said quenching operation in contact with incandescent coke, whereby, reactions are caused to take place between a portion of said water vapor and of the carbon of said coke and a portion of the nitrogen of said coke, with the formation of ammonia; withdrawing from contact with said coke the gases formed in the distilling operation and the gases formed in the quenching operation in a common current; and recovering the ammonia from said gases by contacting the same with a liquid capable of absorbing ammonia, substantially as described.

10. The process of recovering ammonia from coal, which consists, in subjecting said coal to distillation by direct contact with a modified gas flame, said gas flame being modified by admixing with the air current supplied to said flame, a regulated proportion of combustion gases, whereby the temperature of said flame is reduced below the temperature of dissociation of ammonia; passing the products of such distillation in contact with coke from a previous charge of coal, whereby, the products formed in the initial distillation of the coal are in part dissociated with the formation of ammonia and other gases: quenching the coke with water: passing the water-vapor formed in the said quenching operation in contact with incandescent coke, whereby, reactions are caused to take place between a portion of said water vapor, a portion of the carbon of the coke, and a portion of the nitrogen of the coke, with the formation of ammonia; withdrawing from contact with said coke the gases formed in the distilling operation and the gases formed in the quenching operation in a common current; passing said gases through a recuperator, whereby the major portion of their sensible heat is transferred to an air current; conducting said heated air current to said flame to support the combustion in said flame; conducting the cooled gases to an absorbing apparatus and contacting them therein with a liquid capable of absorbing ammonia, substantially as described.

11. The process of recovering ammonia from coal, which consists, in charging said coal onto a column of coke in a coking chamber; subjecting said coal to direct contact with a modified gas flame, said flame being modified by admixing with the air current

supplied to said flame, a regulated proportion of combustion gases, whereby the temperature of said flame is reduced to a temperature not exceeding 1800° F.; passing the products of combustion from said gas flame and the distillation gases from the coal in contact with the coke in, substantially, the upper half of said coke column, whereby, the initially formed products of such distillation are caused to undergo further dissociation with the formation of ammonia and other gases, and the CO_2 and H_2O of said combustion gases are in major part dissociated to CO and H_2; quenching the lower part of said coke column with water; passing the water vapor so formed in contact with the incandescent coke in substantially the lower half of said coke column; withdrawing from said coking chamber the gases formed therein in a common current; passing said gases through a recuperator to heat the air supplied to sustain combustion in said gas flame; conducting the so-heated air to the air passage of the gas burners of said coking chamber; conducting the partially cooled gas to an ammonia scrubber; recovering the ammonia from said gases in said ammonia-scrubber by contacting the said gases with a dilute solution of sulfuric acid; said solution being introduced into said gases as a fog free from droplets of sensible size; recovering such fog from said gases after the ammonia of such gases has been absorbed by said fog by passing the said fog-laden gases through a liquid seal consisting of a liquid containing dilute sulfuric acid; and conducting a portion of the ammonia-free gas back to the gas passages of the gas burners of said coking oven, substantially as described.

Signed at New York city, in the county of New York and State of New York, this 22nd day of Dec. 1909.

HENRY L. DOHERTY.

Witnesses:
THOS. I. CARTER,
W. J. QUENTIN.

Defendants' Exhibit 18.

Patent No. 1,029,528.

(See Opposite ☛)

UNITED STATES PATENT OFFICE.

CARL BOSCH AND HANS KELLER, OF LUDWIGSHAFEN-ON-THE-RHINE, GERMANY, ASSIGNORS TO BADISCHE ANILIN & SODA FABRIK, OF LUDWIGSHAFEN-ON-THE-RHINE, GERMANY, A CORPORATION.

PROCESS OF ABSORBING OXIDS OF NITROGEN.

1,029,528. Specification of Letters Patent. Patented June 11, 1912.

Application filed June 22, 1909. Serial No. 503,637.

To all whom it may concern:

Be it known that we, CARL BOSCH and HANS KELLER, doctors of philosophy and chemists, subjects of the King of Prussia, residing at Ludwigshafen-on-the-Rhine, Germany, have invented new and useful Improvements in Processes of Absorbing Oxids of Nitrogen, of which the following is a specification.

Our invention relates to the absorption of oxids of nitrogen. The absorption of oxids of nitrogen from mixtures containing them (particularly from mixtures obtained by passing air through an electric arc flame) by passing the gases up a tower packed with material down which passes a suspension of a base is open to the objection that the spaces between the packing material gradually become filled with muddy deposit which renders effective absorption difficult and finally chokes up the tower.

According to our invention the absorption of oxids of nitrogen by means of a suspension of a base, or of a basic substance, is effected by introducing a suspension of an absorbent into the gas mixture in a state of fine division, for instance as a spray, or mist. The results obtained thereby are very satisfactory, the gases being better absorbed than they are according to the first-mentioned process, and the process according to our invention being capable of being worked continuously without interruption. In spite of the large quantities of gases which have to be treated, the apparatus required for the purposes of this invention is comparatively small, and less power is required to keep the gases in circulation than is required for the first-named process. As bases which are particularly suitable for use according to this invention, we mention the oxids, hydroxids, and carbonates of the alkaline earth metals and of magnesium. Particularly good results are obtained when using milk of lime.

Of course the process of our invention can be used either for the absorption of the whole quantity of oxids of nitrogen contained in the gases leaving the electric arc flame, or for absorbing the nitrogenous compounds which remain in the gases after they have been partially treated for absorption, for instance after they have been treated with water for the production of nitric acid.

The process of this invention can be carried out in very many different methods without departing from the nature of our invention. For instance, the absorption can be carried out in towers, or in apparatus of any other suitable shape, such for instance as in a number of tubes through which the gases pass in series, or if the tubes be sufficiently large they may be arranged in parallel. If desired, other shaped vessels can be employed. Then again, the suspension of the absorbing agent can be introduced into the apparatus in any suitable manner. As instances of methods which can be used according to our invention, but without in any way limiting ourselves to the methods which we quote here, we mention that the absorbing agent can be introduced through any suitable spraying nozzle, and it can be forced into the apparatus, if desired, by means of compressed air, or, instead of compressed air, the gases containing the oxids of nitrogen can themselves be employed for assisting in the introduction and spreading out into a state of fine division of the absorbing agent.

Instead of using compressed air, or the nitrous gases themselves, other methods can be employed, for instance, the suspension can be made to enter the apparatus by means of its own weight, or it can be forced in by mechanical means. The method of causing the entering, or entered, suspension to assume a state of fine division can also be carried in any suitable manner. For instance, suitable nozzles can be employed, either one or, preferably, more, in each piece of apparatus. The nozzles may allow only the absorbent suspension to enter, or they may be such that at the same time air or other gas enters and assists in spreading out the suspension. The nozzles may remain stationary, or they may have motion imparted to them. If desired, the suspension can enter through the nozzle in the form of a jet and then be subsequently spread out, for instance by causing it to impinge on a suitable surface, either stationary, or in motion, or it

any other suitable manner. The gases, containing, for instance two per cent. of nitric oxid, can be passed into the tower, or other apparatus, either at the top, or at the bottom, or at any other suitable point, or points, and at the same time the suspension, for instance, milk of lime of six degrees Baumé, is passed into the apparatus in any suitable manner, such for instance as hereinbefore described. In this manner an absorption of from ninety eight, to ninety-nine, per cent. of the nitric oxid can easily be effected.

The drawings accompanying this specification represent in vertical section two forms of apparatus suitable for carrying out the process of this invention.

In Fig. 1 C, D represent two towers joined together at the top by the pipe E, the gases enter at the bottom of the tower C, through the opening F, and leave the lower end of the tower D, through the opening G. H H represent suitable nozzles through which the milk of lime is sprayed into the towers, there being several such nozzles in each tower, as represented in the drawing. The milk of lime flows down to the lower end of each tower and through an outlet into the tank B and is thence raised by means of the pump A and again forced through the nozzles H H in the form of spray. This is repeated until the milk of lime has absorbed sufficient of the oxids of nitrogen.

In Fig. 2 the gases are made to pass in a zig zag manner along passages L L which are formed in the tower C by means of partitions K K, arranged after the manner of baffle plates. At the end of each such partition a nozzle H is situated through which milk of lime is sprayed into the space between the partition or the like. In this case the gases enter at the lower end of the tower C and leave at the top, while the milk of lime after being used flows into the tank B and is raised again by the pump A and forced through the nozzles afresh.

A very suitable form of apparatus for use in carrying out the process of our invention consists of a series of tubes or passages arranged in a manner somewhat similar to that shown in Fig. 2, in which the gases are caused to pass through the series of tubes or passages in a zig-zag direction; that is to say, along one passage and then back again in the opposite direction along the next passage, and so on, while at the end of each such tube or passage a nozzle for spraying in the milk of lime is situated. The tubes or passages can be placed side by side, and also one above the other so that they take up a minimum of space. In any case the milk of lime can be sprayed into the tube or passage, either in the direction of motion of the gases or in the opposite direction, as shown in Fig. 2, or any other convenient means of spraying can be followed.

Now what we claim is:—

1. The process of absorbing oxids of nitrogen from mixtures containing them, by introducing a suspension of an absorbent into the gas mixture in a state of fine division.

2. The process of absorbing oxids of nitrogen from mixtures containing them, by introducing milk of lime in a state of fine division into the gas mixture.

3. The process of absorbing oxids of nitrogen from mixtures containing them, by spraying milk of lime into the gas mixture.

In testimony whereof we have hereunto set our hands in the presence of two subscribing witnesses.

CARL BOSCH.
HANS. KELLER.

Witnesses:
ERNEST F. EHRHARDT,
J. ALEC. LLOYD.

Defendants' Exhibit 18.

Patent No. 1,307,534.

(See Opposite ☛)

UNITED STATES PATENT OFFICE.

JOSEPH BECKER, OF PITTSBURGH, PENNSYLVANIA, ASSIGNOR TO THE KOPPERS COMPANY, OF PITTSBURGH, PENNSYLVANIA, A CORPORATION OF PENNSYLVANIA.

AMMONIA, TAR, AND BENZOL RECOVERY PROCESS.

1,307,534. Specification of Letters Patent. Patented June 24, 1919.

Application filed August 12, 1918. Serial No. 249,379.

To all whom it may concern:

Be it known that I, JOSEPH BECKER, a subject of Germany, who have declared my intention of becoming a citizen of the United States and residing in Pittsburgh, in the county of Allegheny and State of Pennsylvania, have invented a new and useful Improvement in Ammonia, Tar, and Benzol Recovery Processes, of which the following is a specification.

My invention relates primarily to the recovery of tar and ammonia, and of benzol, and its homologues, from coke-oven gas, and has for an object a simplified and efficient extraction of the tar, and also a recovery of the ammonia by a method that simplifies apparatus and economizes the heat required in the ammonia recovery; and has for a further object the recovery, directly from the gas, of a portion of benzol, and its homologues, that is carried off dissolved in the tar in the usual methods of tar-extraction from the gas, and the invention may also be applicable to the treatment of gas that is otherwise derived from the carbonization of coal, and may also have such other objects and results as are found to obtain in the processes hereinafter set forth and claimed.

In the accompanying drawings, Figure 1 is a conventional diagram illustrating an embodiment of the invention in a preferred arrangement of apparatus for practising its processes; and Fig. 2 is a key to the symbols employed in the various lines that show the connections between the apparatus indicated in Fig. 1.

5 indicates the coke-ovens from which the coke-oven gas is derived. The gas flows first into an electrical precipitator 6, which it enters at a temperature of about 80° C. At such temperature the tar is carried in the gas in the form of a finely comminuted mist, and such mist is effectively precipitated in the electrical precipitator, from which the precipitated liquefied tar is drained off into the tar-tank 7. The water and ammonia are carried through the precipitator in the form of vapor, as the gas leaves such precipitator at a temperature that is still above the water saturation point of such gas. The gas is then conducted to and enters the hot acid-washer tower 8, in which the ammonia is absorbed by the acid-solution that is kept hot by the absorption reaction. The gas both enters and leaves the acid-washer with a temperature that is still somewhat above its water saturation point, but so little above that practically no water is taken up by the gas from the acid-solution and consequently there is no precipitation of ammonium sulfate in the acid-washer tower and the gas leaves it still carrying as vapor substantially the original water content with which such gas left the ovens. The acid-solution from the said acid-washer tower 8 flows into a saturator 9, carrying with it into the saturator any slight amount of ammonium sulfate that may have been precipitated. The acid-solution drained from the saturator's drain table 10, and centrifugal drier 11, into the mother liquor tank 12, is forced back, by the acid pump 13, to the top of the acid-washer tower 8, a proportion of fresh acid being there continuously added from the acid-tank 14. A portion of the acid-solution from the mother-liquor tank 12 may also flow back into the saturation-bath in the saturator 9. From the acid-washer tower the gas flows into and through the cooler 15 that is sprayed with fresh water from the water tank 16. The drain from this cooler flows to the naphthalene recovery apparatus. The gas issues from the cooler 15 at a temperature of about 30° C., substantially dehydrated, and having been previously freed of its tar and ammonia. Such gas passes next through the exhauster 17 and thence is forwarded in part directly to the saturator, through the gas-line 18, and in part through the by-pass 19 into the gas-line that leads from the saturator to the final cooler 20. The gas passing from the exhauster into the saturator has a temperature of about 35° C., and discharging through the saturation-bath it effects the evaporation requisite to precipitate the ammonium sulfate from the bath. The gas issuing from the saturator, at a temperature of about 45° C., passes, as just above mentioned, to the final cooler 20, together with the gas coming directly through the aforesaid by-pass 19 from the exhauster. This final cooler 20 is constantly sprayed with fresh water from the before-mentioned water-tank 16, and the drain from said cooler is carried to the naphthalene recovery

the naphthalene drain
from the final cooler
still carrying side
original content of ben-
and then passes into
in which the benzol
are extracted by a con-
entering the scrubber
line 25. The benzolized oil
from the scrubber 22 into the benzol
leading to the further appa-
for the recovery of such benzol and its
homologues. The gas from the benzol scrub-
er passes to the gas-holder 26 and thence
its final distribution, in part back to the
ovens to be used as the fuel gas for their
ing, and in part to whatever other service
such clean coke oven gas may be employed
for.

The stripping out of all of the tar while the
gas is still hot, and by a precipitation
method that does not require the gas to pass
through any scrubber or series of tar
over the absorption of benzol and its
homologues that occurs when the gas
other cooler to effect the extraction of
vapor through a series of liquid
and there with a substantial portion
of the ammonium sulfate precipitated in
saturator with gas that has not undergone any scrubbing operation other than the
stripping incidentally derived from the solu-
tion of ammonium sulfate which is deposited in
passing through the saturator. The naph-
thalene is removed with the drainage from
the first cooling operation to which the gas
is subjected, that is, the cooling operation is
subsequent to both the tar precipitation and
the ammonia absorption, both of which are
effected while the gas is still at high tem-
perature; and so naphthalene stoppages in
the apparatus are effectively avoided.

The processes of the invention may be
practised in various ways, other than the
particular embodiment that has been de-
scribed for purposes of illustration, and fall
within the scope and subject matter of the
claims hereinafter made.

In my co-pending application Serial No.
[illegible], filed [illegible] date, I have set forth and directed
claim to the general process of gleaning
the stripping from the hot gas by passing it
through an acid wash and then through a
hot ammonia-charged solution to a
saturation bath through which the
monia freed and charged gas is passed and
gas is passed under conditions of
from such bath.

I claim:

1. In a process for the recovery of tar and ammonia from coke oven gas, the combination of steps that consists in extracting the tar from the coke oven gas hot; passing such hot gas through an acid wash to absorb the ammonia of the gas; discharging the mother liquor from such acid wash into a saturation bath; passing the tar free and ammonia free gas through a cooling and dehydrating operation; and passing such gas through said saturation bath, to effect deposition of the ammonium sulfate; substantially as specified.

2. In a process for the recovery of benzol and its homologues from coke oven gas, the combination of steps that consists in electrically precipitating the tar residue from the gas while the latter is still hot enough to preclude appreciable absorption of benzol by the tar precipitated; and then passing the gas through ammonia recovery and benzol extraction operations; substantially as specified.

3. In a process for the recovery of tar, ammonia and benzol, and its homologues, from coke oven gas, the combination of steps that consists in electrically precipitating the tar residue from the gas while the latter is still hot enough to preclude appreciable absorption of benzol by the tar precipitated; passing the gas through an acid solution at a temperature above the water saturation point of the gas but low enough to preclude substantial deposition of ammonia sulfate in said solution; separating said ammonia-charged solution into a saturation bath; dehydrating said ammonia freed gas and then passing it through said saturation bath to effect deposition of the ammonium sulfate; and then passing said gas to the further operations for the recovery of benzol and its homologues; substantially as specified.

4. In a process for the recovery of benzol and its homologues from benzol charged gas, the combination of steps that consists in electrically precipitating the tar residue from the gas while the latter is still hot enough to preclude appreciable absorption of benzol by the tar precipitated; and then passing the gas through the further operations relevant to the recovery of the benzol and its homologues; substantially as specified.

5. In a process for the recovery of tar, ammonia and benzol, and its homologues, from coke oven gas charged with them, the combination of steps that consists in electrically precipitating the tar residue from the gas while the gas is enough superheated substantially to preclude the tar from absorbing the benzol and its homologues, in the gas; passing the freed gas through an ammonia ab-

sorbing acid-solution at a temperature above the water-saturation point of the gas but low enough to preclude substantial deposition of ammonium sulfate in said solution: conducting said ammonia-charged solution into a saturation-bath: dehydrating said ammonia-freed gas and then passing it through said saturation-bath to effect deposition of the ammonium sulfate: and then passing said gas to the further operations for the recovery of benzol and its homologues: substantially as specified.

In testimony whereof, I hereunto set my hand in the presence of two subscribing witnesses.

JOSEPH BECKER

Witnesses:
HENRY LOVE CLARKE,
JOSEPH DVORAK.

Defendants' Exhibit 18.

Page 220 of Hutte's Book.

(See Opposite ☛)

c) Naßreiniger.

1. Rieselwascher.

Rieselwascher (Skrubber) ohne Einbauten (Abb. 12). Bis 20 m hohes zylindrisches oder eckiges Gehäuse aus Eisen oder Beton. Gas von unten nach oben mit rd. 0,5 m/sk Geschwindigkeit. Im Gegenstrom durch Streudüsen o. ä. Waschfl. eingespritzt. — Genügt zur Abscheidung groben oder hygroskopischen Staubes oder zur Durchführung schnell verlaufender Rkk. und Absorptionen. Einfache Bauart, großer Platz-, geringer Kraftbedarf, ständige Pumpenleistung.

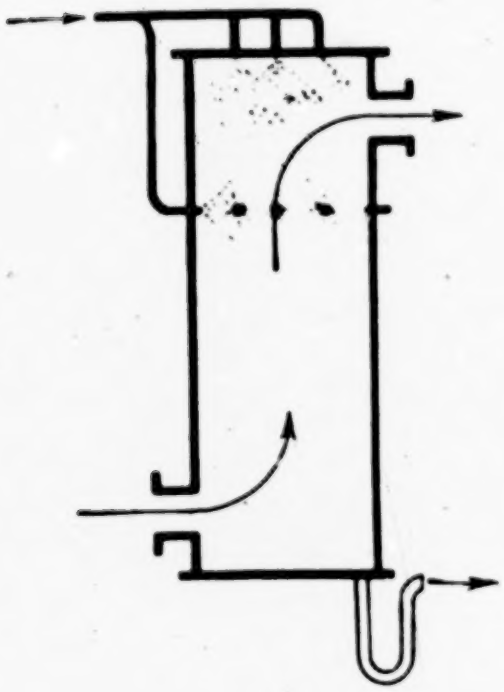


Abb. 12.

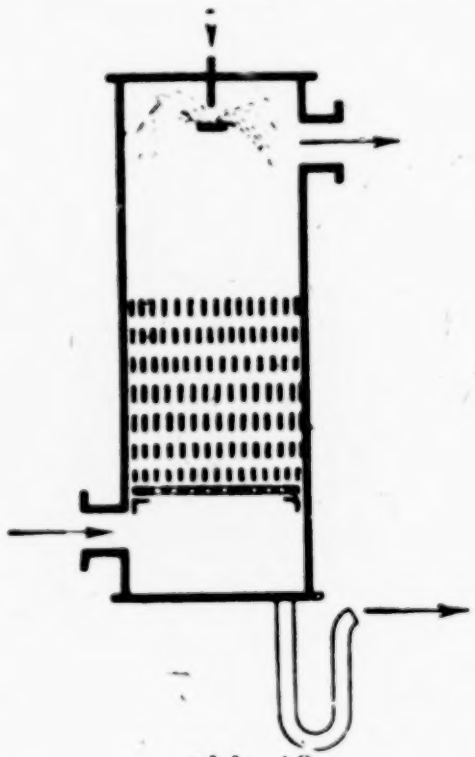


Abb. 13.

Rieselwascher mit Einbauten (Abb. 13) liefern bessere Ergebnisse. Türme niedriger, weil Horden, Füllringe (z. B. Raschig), Koks u. dgl. Waschfl. und Gas länger und besser in Berührung bringen. Sehr gute Verteilung der Fl. durch die besonders ausgebildeten Horden der Zschocke-Werke in Kaiserslautern, wenn man nicht zu Füllringen greifen will. Mit Horden für ziemlich stark staubhaltige Gase verwendbar (mit Füllringen für staubarme). Bei sehr unreinem Gas (über 5 bis 7 g/m³) vor Zentrifugalwascher geschaltet.

Im ungefüllten Querschnitt soll die Gasgeschwindigkeit 0,5 m sk nicht übersteigen. Bei Füllung mit Raschigringen von 25 mm Dm. können auf 1 m², ohne den Druckverlust von 5 mm auf 1 m Schütthöhe bei 0,5 m/sk Geschwindigkeit zu erhöhen, bis 2 m³ W./st aufgegeben werden. Bei 6 m³/st Druckverlust etwa 10 mm W.S.; bei 10 m³/st Gefahr von Stauungen. Von zäher Waschfl. ist die Menge entsprechend herabzusetzen. Reinigung befriedigend. Unter Umständen müssen mehrere Türme hintereinander, nicht parallel, aufgestellt werden. — Platzbedarf groß, Kraftbedarf hauptsächlich für Pumpenleistung. Auch als Kühler sehr gut wirksam.

Excerpt From Defendant's Exhibit 18.

HUTTE

HANDBOOK

FOR THE

PRACTICAL CHEMIST (1927)

c) Wet Purifiers

1. Spray Scrubbers

Spray Scrubber without obstructions (Fig. 12). Up to 20-meter. high cylindrical or angular housing made of iron or concrete. Gas from bottom to top at a velocity of about 0.5 m/sec. Scrubbing liquor is sprayed counter-currently through spray nozzles or the like. Satisfactory for separation of coarse or hygroscopic dusts or for carrying out rapidly-proceeding reactions and absorptions. Simple construction, large space requirement low power consumption, constant pumping output.

Spray Scrubber with baffles (Fig. 13) produces better results. Towers are lower since hurdles, packing rings (e. g. Raschig), coke, and the like effect longer and better contact between gas and liquid. Very good distribution of the liquid by the specially constructed hurdles of the Zschocke-Werke in Kaiserslautern, when one does not want to use packing rings. Usable with baffles for gases containing large amounts of dust (with packing rings for gases containing little dust). In the case of very impure gases (above 5 to 7 g/m^3), connected before centrifugal washer.

The gas velocity should not exceed 0.5m/sec. in the unpacked cross-section. When packed with Raschig rings of 25 mm diameter it is possible to use from 1m3 to 2m3 of washing liquid per hour, without increasing the pressure loss of 5 mm with 1m packing height at 0.5 m/sec. velocity. At $6m^3$/hr. pressure loss about 10 mm. water column; at $10m^3$/hr. there is danger of obstruction. The amount is correspondingly lowered in case of viscous scrubbing liquor. Under certain circumstances several towers must be installed one after another but not parallel. Space requirement high; power requirement mainly for pumping. Also very effective as a cooler.

Defendants' Exhibit 19.

(See Opposite ☛).

H. L. DOHERTY

PROCESS OF OBTAINING AMMONIA FROM COAL

997,908

Patented July 11, 1911

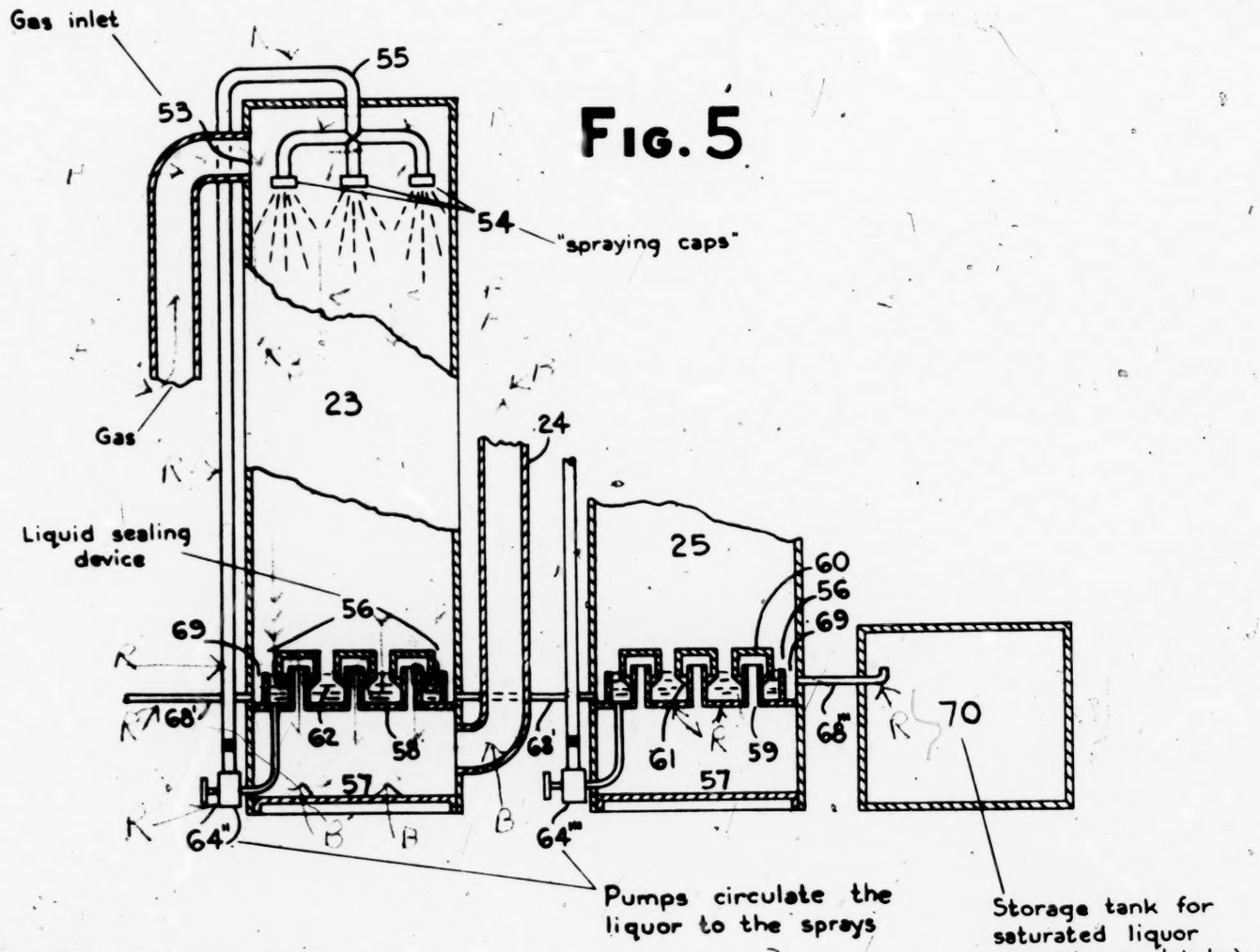


FIG. 5

R Red indicates liquor.
B Blue indicates gas.

"In passing through [the absorption apparatus] the gases are cooled and the ammonia which they contain is absorbed by a suitable liquid, ['a dilute solution of sulphuric acid'] which is circulated through the tower."

(P. 2, Col. 1, last line, et seq.)

The spray heads produce "a very fine spray."

(P. 2, Col. 2, l. 112.)

"It is apparent, that my method of absorbing the ammonia insures the most intimate contact possible between the gas and the liquid, the absorption of the ammonia being rapid and complete. I thus reduce the capacity necessary in the absorbing apparatus to a minimum."

(P. 3, Col. 1, l. 64, et seq.)

[Emphasis added.]

Def's. Ex. 19

Defendants' Exhibit 20.

(See Opposite ☛)

C. BOSCH & H. KELLER

PROCESS OF ABSORBING OXIDS OF NITROGEN

1,029,528

Patented June 11, 1912

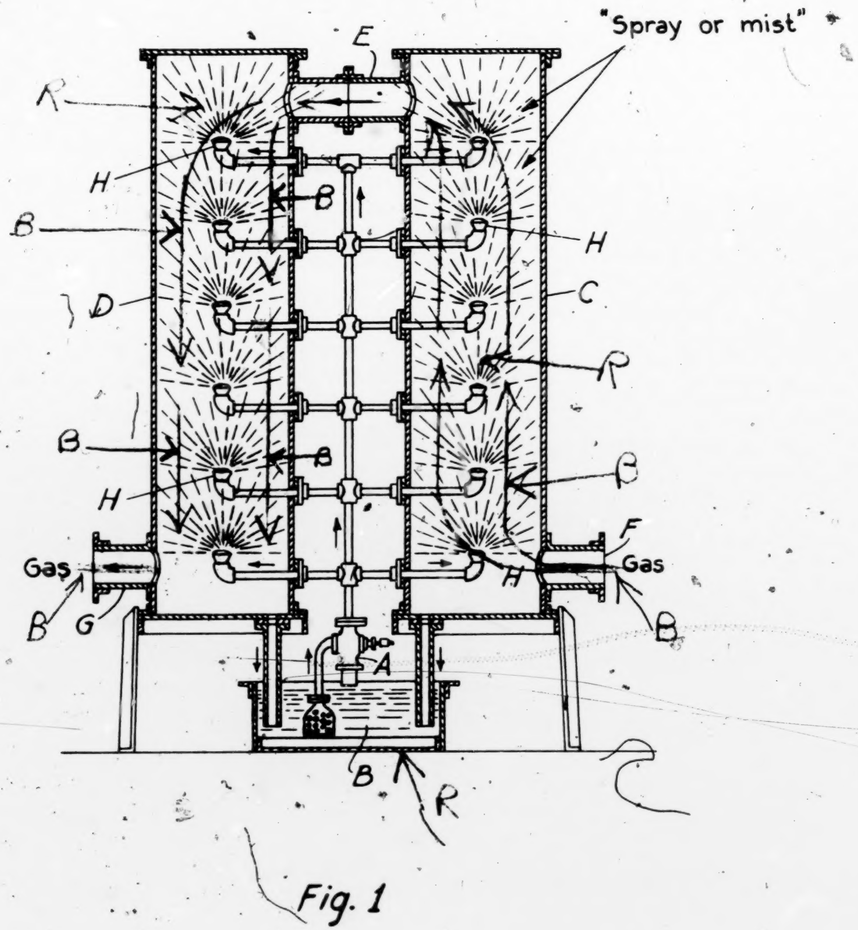


Fig. 1

Red indicates liquor.

"In spite of the large quantities of gases which have to be treated,

(1) the apparatus required for the purposes of this invention is comparatively small, and

(2) less power is required to keep the gases in circulation. . ."

(Col. 1, lines 34-37)

Def's. Ex. 20

Defendants' Exhibit 21.

(See Opposite ☛)

J. BECKER

AMMONIA, TAR AND BENZOL RECOVERY PROCESS.

APPLICATION FILED, AUG. 12, 1918.

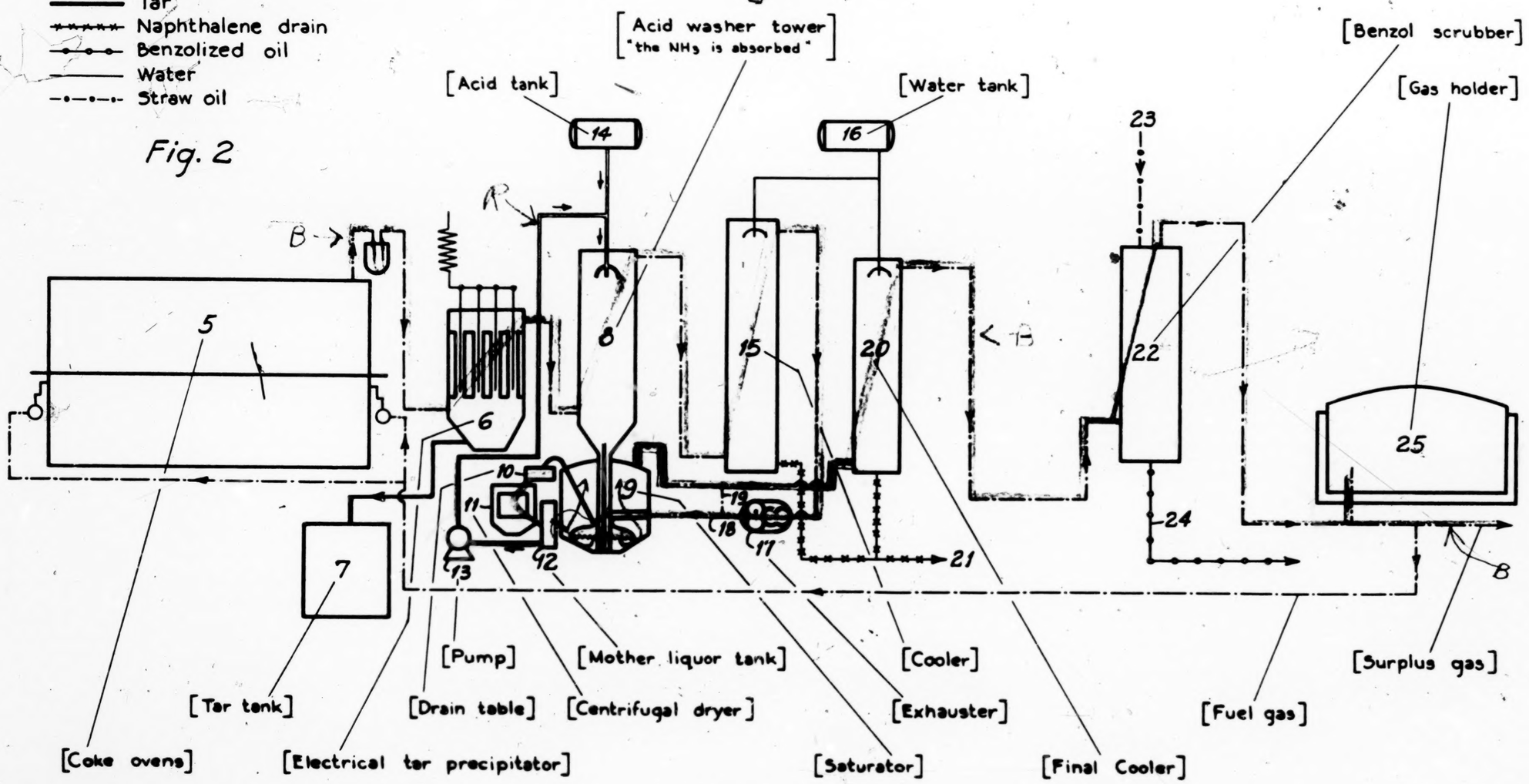
1,307,534.

Patented June 24, 1919.

Coke oven gas
Acid
Tar
Naphthalene drain
Benzolized oil
Water
Straw oil

Fig. 2

Fig. 1



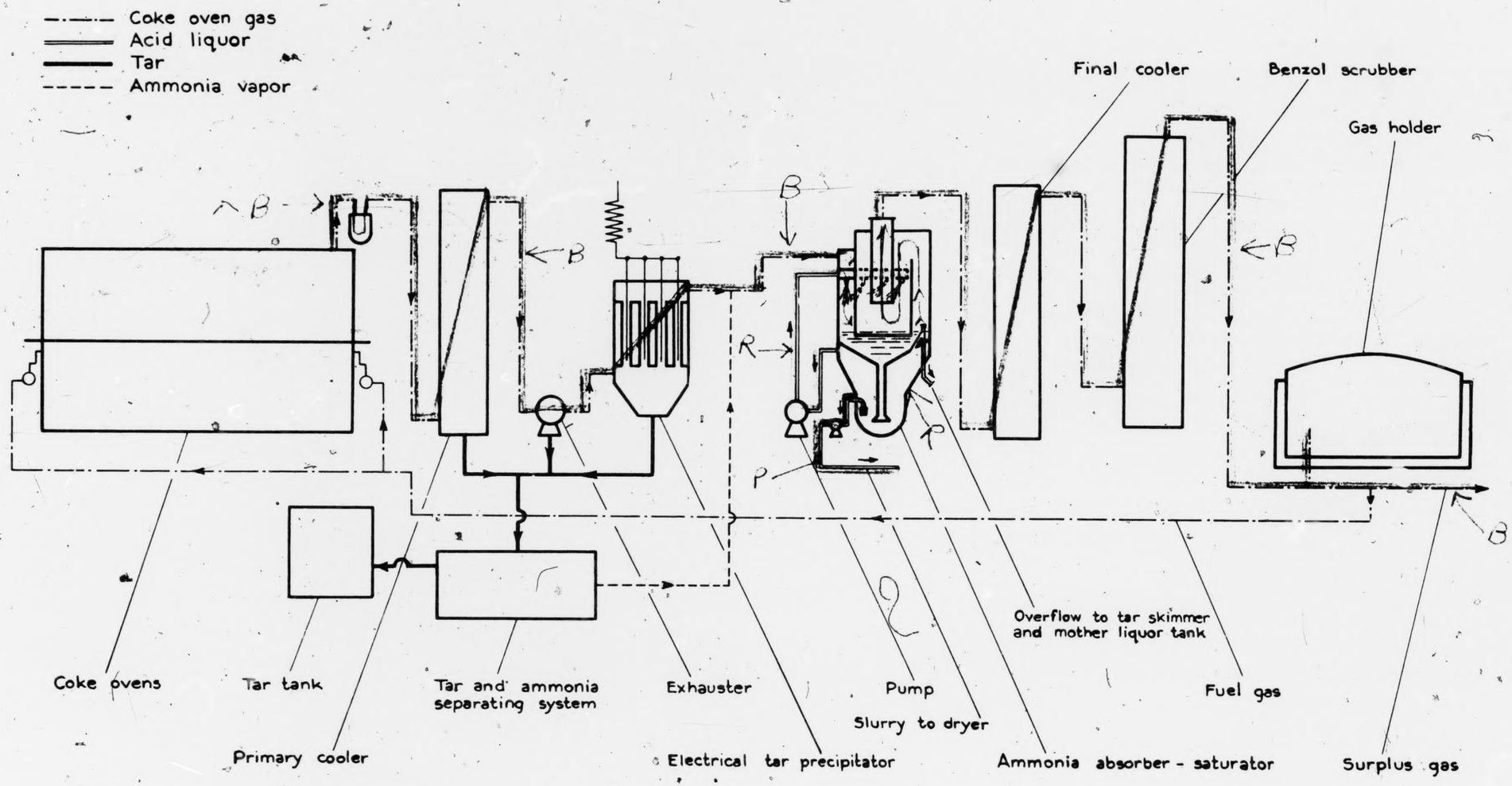
Red indicates liquor

Def's. Ex. 21

Defendants' Exhibit 22.

(See Opposite ☛)

WHEELING INSTALLATION



Red indicates liquor.
Blue indicates gas.
Purple indicates slurry.

Def's. Ex. 22

Defendants' Exhibit 23.

(See Opposite ☞)

HÜTTE HANDBOOK

PUBLISHED 1927

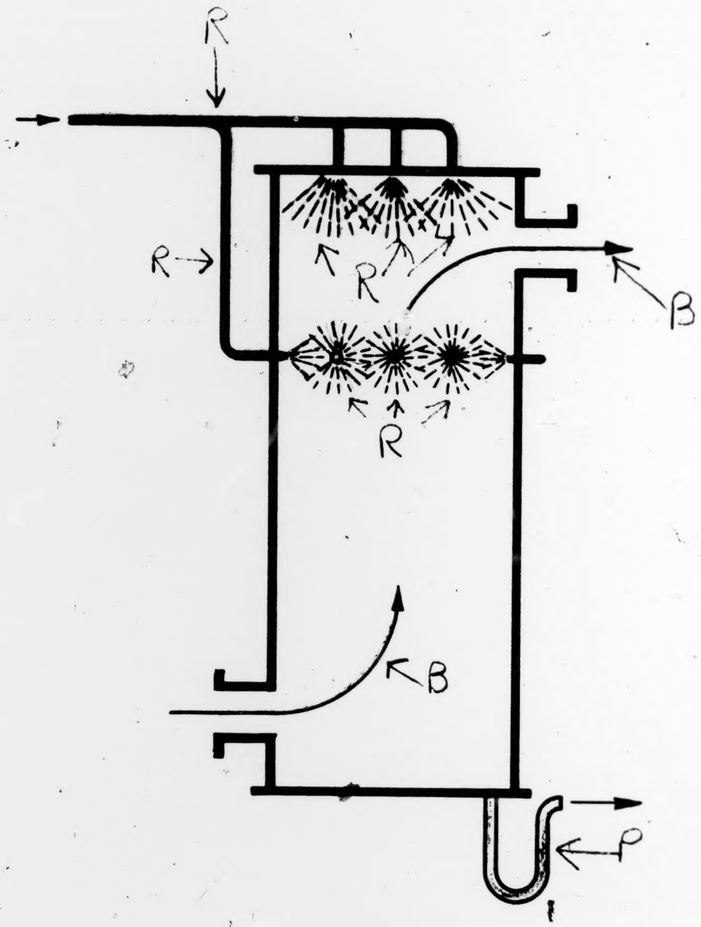


Abb. 12
[Fig. 12]

Red indicates liquor.
Blue indicates gas.
Purple indicates slurry.

Spray Scrubber without obstructions (Fig. 12)...Scrubbing liquor is sprayed countercurrently through spray nozzles or the like. Satisfactory for separation of coarse or hygroscopic dusts or for carrying out rapidly-proceeding reactions and absorptions. Simple construction, large space requirement, low power consumption, constant pumping output. (P. 220, emphasis added.)

Def's. Ex. 23

Defendants' Exhibit 24.

(See Opposite ☛)

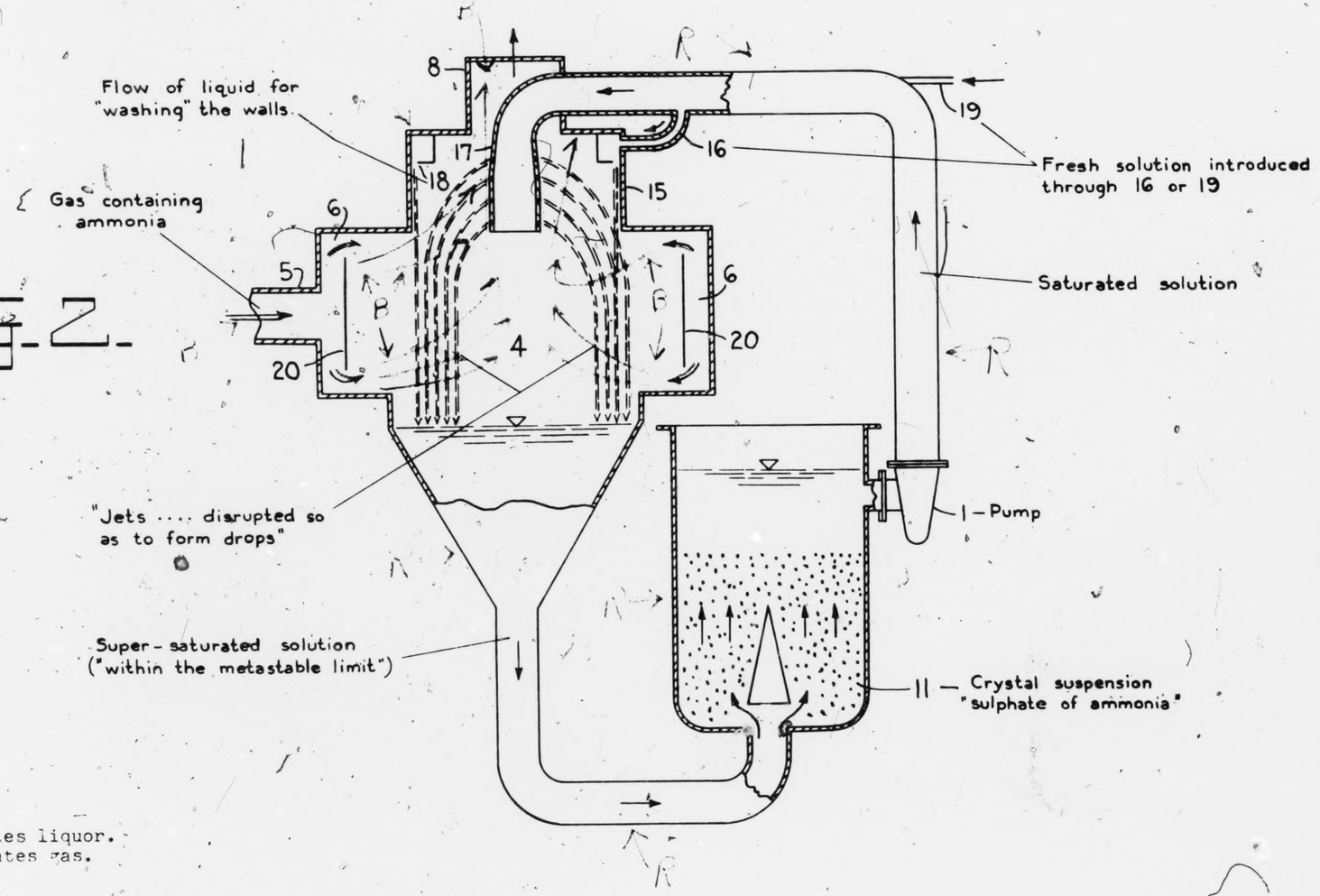
F. JEREMIASSEN

TREATMENT OF SOLUTIONS TO OBTAIN SOLID MATTER THEREFROM

2,375,922

Patented May 15, 1945

Fig. 2.



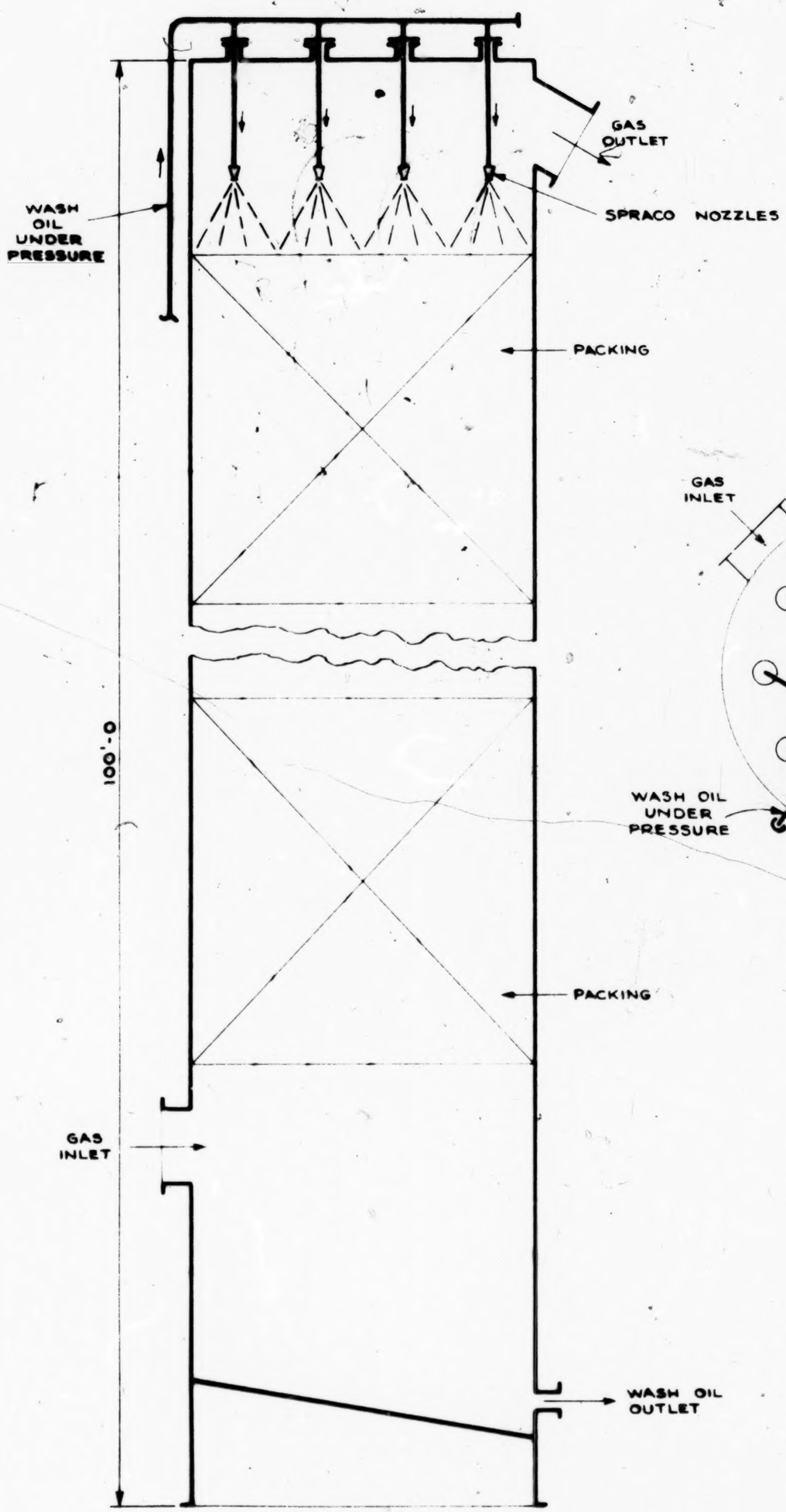
Red indicates liquor.
Blue indicates gas.

Def's. Ex. 24

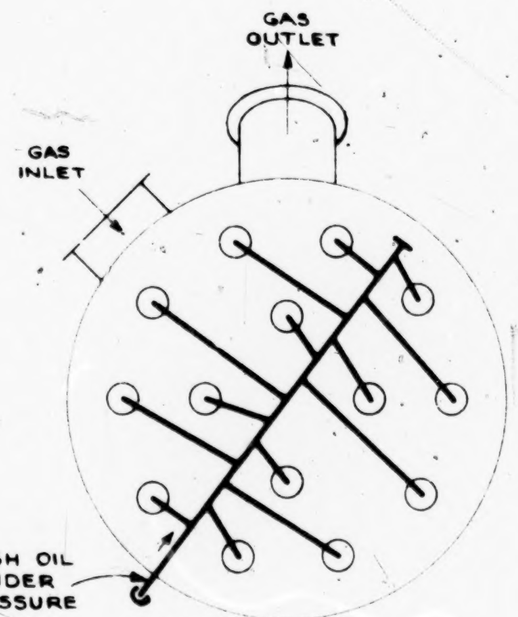
Defendants' Exhibit 26.

(See Opposite ☛)

Def's. Ex. 26



SECTION



PLAN

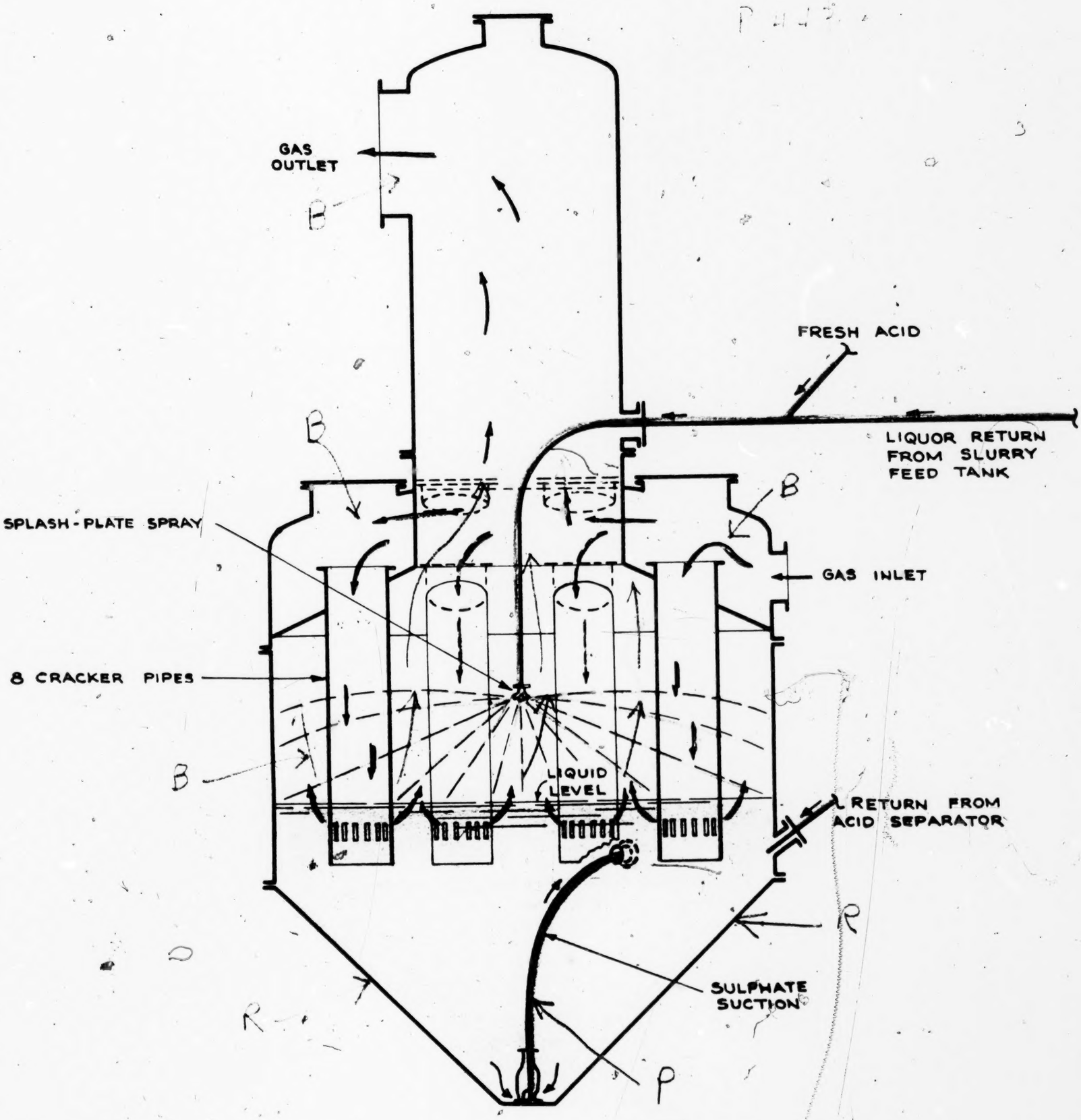
Red indicates wash oil.
Blue indicates gas.

BENZOL WASHER
WITH SPRACO NOZZLES
(USED SINCE 1929)

Defendants' Exhibit 27.

(See Opposite ☞)

Def's. Ex. 27

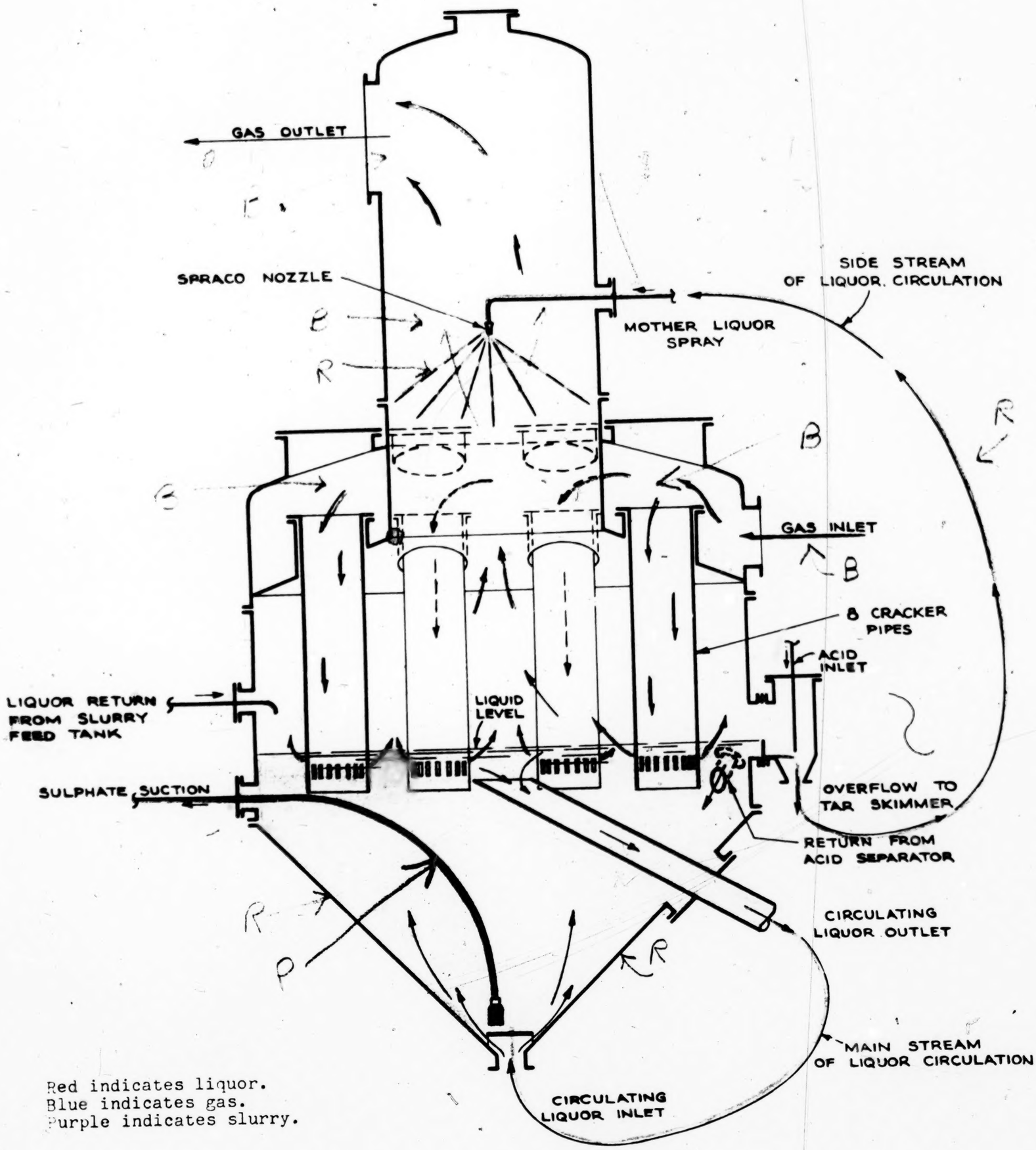


Red indicates liquor.
Blue indicates gas.
Purple indicates slurry.

MONESSEN SATURATOR
(1943)

Defendants' Exhibit 28.

(See Opposite ☛)



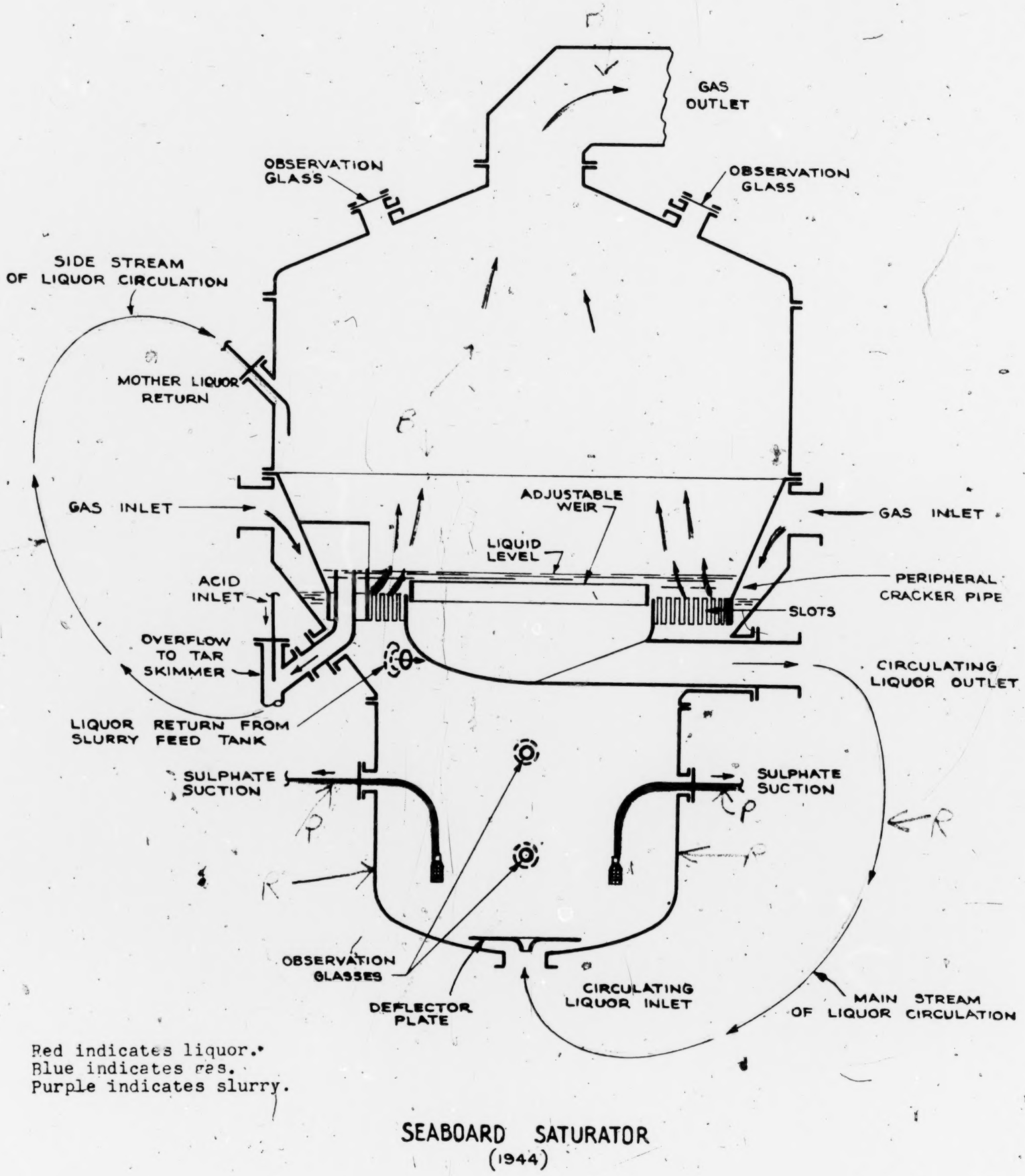
Red indicates liquor.
Blue indicates gas.
Purple indicates slurry.

WEIRTON SATURATOR
(1946)

Defendants' Exhibit 29.

(See Opposite ☛)

Def's. Ex. 29



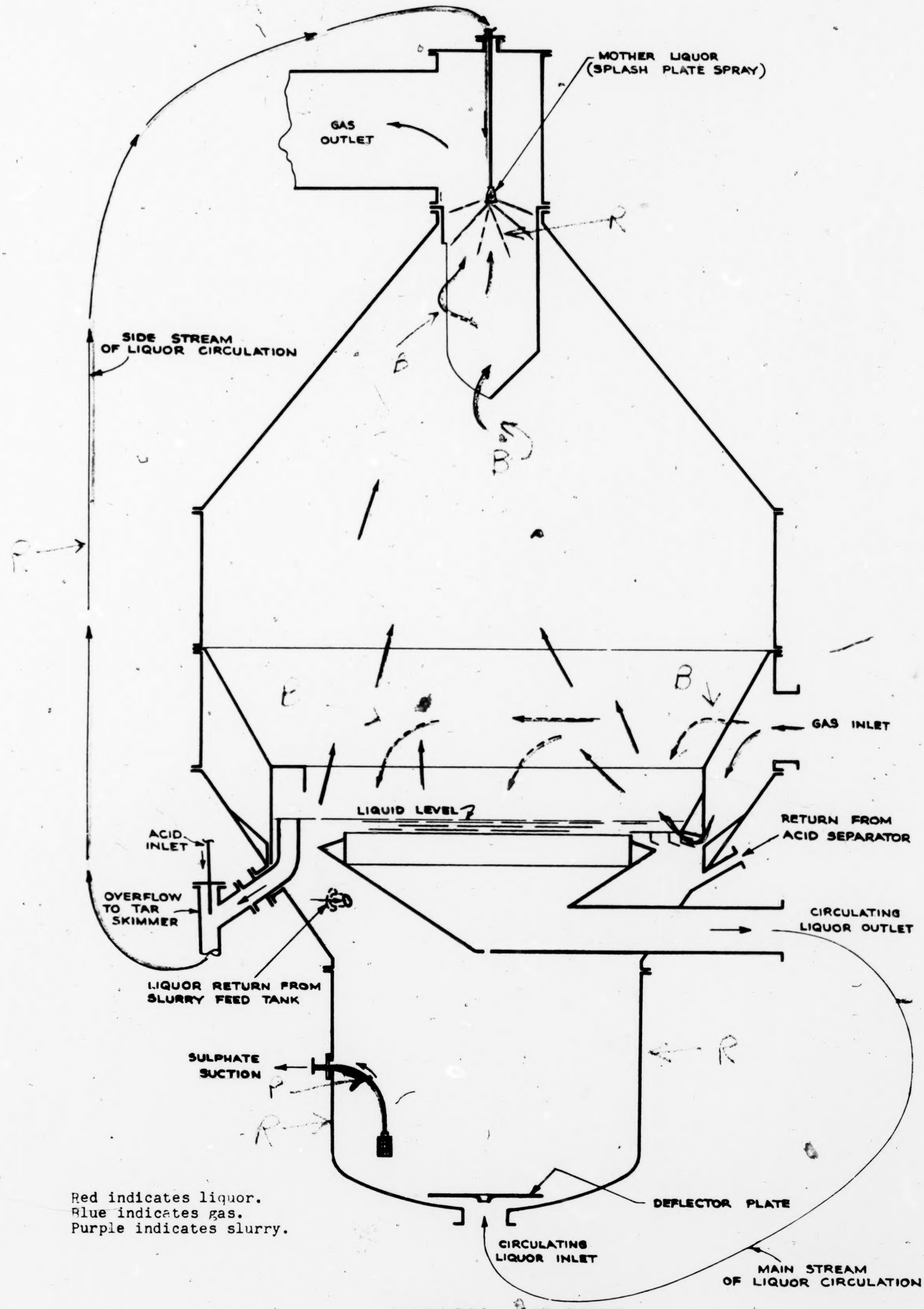
Red indicates liquor.
Blue indicates gas.
Purple indicates slurry.

SEABOARD SATURATOR
(1944)

Defendants' Exhibit 30.

(See Opposite ☛)

Def's. Ex. 30



Red indicates liquor.
Blue indicates gas.
Purple indicates slurry.

ALIQUIPPA SATURATOR
(MAY, 1947)

Defendants' Exhibit 37.

(See Opposite ☛)

Patented Apr. 16, 1935 1,997,757

UNITED STATES PATENT OFFICE

1,997,757

SATURATOR FOR THE PRODUCTION OF AMMONIUM SALTS AND PROCESS FOR MAKING SUCH SALTS

William Tiddy, Scarsdale, N. Y., assignor to Semet-Solvay Engineering Corporation, New York, N. Y., a corporation of New York

Application August 15, 1931, Serial No. 557,344

15 Claims. (Cl. 23—119)

This invention relates in general to a process and apparatus for mixing gaseous and liquid media, and in particular to a process for the absorption of ammonia from ammonia-containing gases, and an improved spray-roll saturator for the same.

The preparation of ammonium salts, particularly of ammonium sulfate in crystalline form as heretofore carried out has been attended with numerous difficulties. Heretofore the complete absorption of ammonia gas in liquid media, particularly acids, has been difficult due to the temporary formation of saturated layers at the interface of liquid and gas, a condition retarding the rate of solution of the gas. A further disadvantage of known types of ammonia saturators resides in the fact that the salt crystals produced in such satufators are usually small in size. The small size of the crystals hinders their complete separation from the mother liquor. Further, a mass of small crystals presents such a large surface area to washing fluids that a substantial amount of the crystalline mass is lost in purification processes involving washing. Further, the removal of mother liquor from a mass of small crystals by centrifuging is obviously accomplished only with the expenditure of much time and power and frequently with the loss of a substantial amount of the crystalline mass.

It is an object of this invention to provide an apparatus which shall effect the substantially complete removal of ammonia from gas containing the same by contacting such gases with a spray of acid produced by rotating a cylindrical surface in tangential contact with the surface of a body of an acid liquor.

Another object of this invention is to provide an apparatus designed to effect the production of ammonium salts in crystalline form and to promote the growth of the salt crystals in order to facilitate the separation of such crystals from the mother liquor.

Another object of this invention is to provide an apparatus for the recovery of ammonia from gas which is designed to prevent the establishment of back-pressure on the stream of gas

It is a further object of this invention to provide a process for the production of ammonium salts from an ammonia-containing gas and a fine spray of a solution containing an acid.

It is an additional object of this invention to provide a process for preparing ammonium sulfate in the form of relatively large crystals, which may be easily and substantially completely separated from the mother liquor

In accordance with my invention an acid is dispersed as a fine spray by revolving a cylindrical surface about a substantially horizontal axis and substantially in tangential contact with the surface of an acid liquid. The spray of finely dispersed liquid may be contacted with a suitable ammonia-containing gas in such a manner that the sprayed liquid containing dissolved ammonium salts is continuously returned to the main body of liquid in the reservoir. The liquid reservoir is of such dimensions that the crystals of the ammonium salt, after their formation, are maintained in contact with the extended body of liquor saturated with the ammonium salt for a relatively long period of time, thus effecting a substantial growth of the crystals and facilitating their subsequent separation from the mother liquor.

I have discovered that ammonium sulfate crystals of large size may be produced by the use of my improved ammonia saturator which effects the absorption of ammonia from ammonia-containing gases in a solution containing sulfuric acid and the maintenance of the ammonium sulfate thus produced in contact with an extended body of sulfuric acid saturated with ammonium sulfate for a long period of time.

For a fuller understanding of the nature and objects of the invention, reference should be had to the drawing accompanying this specification, showing one form of my improved ammonia-saturator, in which,

Fig. 1 is a side elevation of my improved spray apparatus taken on the line 1—1 of Fig. 2; and

Fig. 2 is an end elevation and section of the spray apparatus taken on the line 2—2 of Fig. 1.

In the drawing, with special reference to Fig. 2, my improved apparatus comprises a casing 1 formed by downwardly converging end walls 3 of substantial length, side walls 4 also of substantial length, one of which side walls may converge toward the other to form a bottom 5, the end walls 3 and side walls 4 defining a relatively high narrow chamber for maintaining an extended body of liquid therein as more fully described hereinafter. The casing is provided with a cover member 6 adapted to be readily removed to allow access to the interior of the casing, the cover member defining a spray chamber 8 disposed directly above casing 1. An inlet 7 is provided in one end of chamber 8 for gas entering the spray chamber, and an outlet 9 is provided in the opposite end for the exit of the gas after its passage through the spray chamber. Spray rolls 10 are rotatably mounted adjacent the bot-

tom of the spray chamber 8 and the top of the body of liquor in container 1. Shaft 13 of the spray roll, positioned substantially horizontal and journaled in suitable bearings 12 and passing through bearing sleeve 14 provided with packed gland 15, are driven by any suitable means, such as by electric motors 16 the armature shafts of which may be integral with or connected to the shafts 13 of spray rolls 10.

A body of liquid, such as an absorbent, is maintained in reservoir 17 at a constant level by known means, such as an overflow device 11 communicating with the body of liquid in the reservoir 17 at a point below the horizontal axes of the spray rolls. The surface of the liquid in container 1 is thus maintained at a substantially constant level in tangential contact with the spray rolls. By maintaining the surface of the rolls in tangential contact with the surface of the liquid in container 1 a spray above the liquid is formed without substantial agitation of the body of the liquid.

At the bottom of the casing means for discharging the absorbent containing absorbed and suspended bodies may comprise, for example, inlet 19 carrying a steam or vapor injector pipe 21 and an outlet 20 carrying a discharge pipe 22. Discharge pipe 22 communicates with a sludge box or tank (not shown) which separates the crystals from the mother liquor. The recovered absorbent or mother liquor is preferably returned to the body of absorbent near the top thereof, or it may be returned through the spray chamber, for example, by means of inlet 23. The inlet for fresh acid or mother liquor containing the same is so positioned with respect to the spray rolls that the fresh acid is sprayed as quickly as possible after being charged into the reservoir and before substantial dilution of the fresh acid by the liquor contained in the reservoir has occurred. The apparatus of my invention may be made of any suitable corrosive-resistant material or of corrosible material if such material is protected by means of a suitable lining 18 of non-corrosible material, for example, lead.

While my invention contemplates an apparatus comprising a plurality of cylinders positioned horizontally with respect to their longitudinal axes and arranged in parallel, in combination with a reservoir of relatively large dimensions, it will be obvious that my apparatus may comprise a single cylinder in combination with such a reservoir. The cylinder may be of any desired dimension but I prefer to use a cylinder having a diameter of about 6 inches. The rotatable cylinder may be perforated, in which case I prefer to employ a hollow cylinder perforated with a large number of relatively small holes. Or, if desired, a rotatable cylinder made up entirely of wire screen or a roll composed of a plurality of discs may be employed in my improved saturator. I prefer to use an independent driving element for each cylinder, however, I may employ a single driving element to operate a plurality of cylinders.

While a spray chamber of any desired shape and dimension may be employed in combination with a suitable rotatable cylinder, I prefer to employ a spray chamber or hood having an arched or curved ceiling and of a width substantially equal to the length of the cylinder and of such height that, when the cylinder is throwing a uniform spray the spray will completely fill the spray chamber. The curved or arched ceiling of the spray chamber promotes the quick return to the reservoir of the sprayed liquid

I have found that the growth of crystals is promoted if a liquid reservoir is employed of such dimensions that an extended body of liquid may be contained in the same. While efficient growth of crystals according to my improved process may be achieved by having the reservoir extended primarily with respect to either a horizontal axis or a vertical axis, I prefer to employ a reservoir having substantial extension with respect to a vertical as well as to a horizontal axis.

In the preferred form of my apparatus the discharging means associated with the acid reservoir and adapted to discharge an acid liquor containing suspended salt crystals is so positioned with respect to the point of entry or formation of the ammonium salt crystals in the acid liquor that crystals passing to the discharging means through the body of acid liquor undergo a substantial growth before being discharged from the reservoir. Furthermore, as appears from Figs. 1 and 2, the mechanism for effecting discharge of the crystals is disposed to one side (to the right, viewing Fig. 2) of the deep body of acid-containing liquid. Hence, steam or vapor blown through ejector pipe 21 to effect the discharge of the crystals will not cause substantial agitation of the deep body of acid-containing liquid.

By removing the solid reaction products substantially at the base of the reservoir, the crystals are thus permitted to remain in contact with a solution of acid saturated with the same for an extended period of time. Crystals produced in the spray and in the upper portion of the liquid body are usually of relatively small dimensions, however, by the time such crystals have reached the discharge means associated with the reservoir, they are found to be materially increased in size due to long continued contact with the saturated mother liquor, which as hereinabove pointed out is not agitated either by the spray producing rolls or the crystal discharge mechanism. Accordingly my improved apparatus is particularly applicable for the production of salts in a solid form, comprising crystals of relatively large size.

As an example of the preferred modification of the process of my invention and of the operation of my improved saturator, reference should be had to the following detailed example.

For the preparation of ammonium sulfate from ammonia-containing gas and a liquid containing sulfuric acid, reservoir 17 may be filled with an aqueous solution of sulfuric acid and the level of the liquid maintained substantially constant and in tangential contact with the hollow perforated rolls 10 by feeding fresh sulphuric acid, mother liquor, or both to the reservoir in amounts somewhat greater than the amount of liquor sprayed by the spray rolls into the gas stream and withdrawn with the crystals from the base of reservoir 17. The cylinders are rotated at such a speed that the acid liquid is continuously carried out of the body of liquid and centrifugally discharged from the cylinder as a fine spray completely filling the annular space enclosed by the spray chamber 8. An ammonia-containing gas, for example coke oven gas or other gas containing the same, may be introduced into the spray chamber by means of gas inlet 7 and contacted with the spray. Due to the extended gas-liquid interface existing in the mixture of spray and gas, the absorption of ammonia proceeds rapidly to completion. The resulting ammonia-free gases may be exhausted from the spray chamber through nozzle 9. The reaction of the ammonia with sulfuric acid and water in the sprayed liq-

uor results in the formation of ammonium sulfate. The sprayed liquor containing ammonium sulfate, both dissolved and crystalline, drips down the side of the spray chamber and is returned to the main body of liquid in the reservoir, which liquid thus becomes slowly saturated with ammonium sulfate. The ammonium sulfate crystals, after formation, remain in contact with the saturated solution of the same for a long period of time owing to the extended body of liquid provided by the use of the large reservoir 17 and further owing to the position of the discharge means associated with the reservoir. This condition results in a substantial growth of the crystals of ammonium sulfate. The solution of sulfuric acid containing ammonium sulfate, both as dissolved and solid salt, may be discharged from the base of the reservoir, for example, by ejecting the solution through pipe 22 by blowing in steam through pipe 21.

The liquor is then passed to a suitable separating device, for example, a centrifuge, wherein the crystalline ammonium sulfate is freed from liquor. The centrifuged liquor may be mixed with fresh sulfuric acid to restore the concentration of sulfuric acid to its original value and the concentrated liquor returned to the reservoir at any convenient point, preferably through an inlet provided near the base of the spray rolls 10 or through inlet 23 provided in the top of the spray chamber C.

The process of my invention likewise is adapted to the production of ammonium salts other than ammonium sulfate. For example, by charging reservoir 17 with a solution containing phosphoric acid and passing an ammonia-containing gas through the spray chamber. I may prepare ammonium phosphate substantially as in the manner above described for the preparation of ammonium sulfate.

Further, the invention herein disclosed is applicable to the production of salts other than ammonium salts from the basic and acid constituents thereof, by contacting one of the constituents of said salt in the gaseous phase with a spray of a liquid containing the other constituents. For example, I may employ a gas containing acid anhydrides such as sulfur dioxide, carbon dioxide, etc. and contact such a gas, which may be a coke oven gas, with a spray of an aqueous solution of an alkaline compound such as sodium hydroxide.

It will be observed that when a flowing stream of gas is passing through the spray chamber of my improved saturator, the stream is contacted with a fine spray of acid which establishes substantially no resistance to the flow of the gas. Accordingly the apparatus of this invention may be advantageously associated with a gas generator or coke oven and used for the recovery of gaseous constituents from the make gases thereof. The use of this apparatus obviates the difficulties of forcing a gas stream through a liquid seal as required in bell or other known saturators.

Since certain changes in carrying out the above process and in the constructions set forth which embody the invention may be made without departing from its scope, it is intended that all matter contained in the above description and shown in the accompanying drawing shall be interpreted as illustrative and not in a limiting sense.

I claim

1. In an ammonia saturator, the combination with a spray-roll for producing a fine spray of liquid and a spray chamber enclosing said roll and arranged to contact an ammonia-containing gas with said spray, of a liquid reservoir and means associated with said reservoir for discharging liquid containing suspended crystals therefrom, said reservoir having dimensions such that crystals charged into the upper portion of a body of liquid contained in said reservoir will travel through an extended body of liquid before being discharged therefrom.

2. In an ammonia saturator, the combination which comprises a liquid reservoir, a spray chamber positioned above said reservoir, a spray roll rotatable in tangential contact with a body of liquid contained in said reservoir, means for passing a gas through said spray chamber and discharging means positioned in said reservoir at such a point that crystals produced in the liquid contained in said reservoir will undergo a substantial growth before being discharged therefrom.

3. In an ammonia saturator, the combination which comprises an acid reservoir, a spray chamber positioned above said reservoir, a cylinder positioned in tangential contact with liquid contained in said reservoir, means for revolving said cylinder at such a speed that acid contained in said reservoir will be dispersed as a fine spray, means for passing ammonia in contact with said spray, and means for discharging acid containing suspended crystals from said reservoir, said discharging means positioned in said reservoir at such a point that crystals of an ammonium salt produced in said reservoir will undergo a substantial growth before being discharged therefrom.

4. In an ammonia saturator, the combination which comprises a relatively deep acid reservoir, a spray chamber positioned above said reservoir arranged to direct sprayed liquid downward, a hollow perforated cylinder rotatable about a horizontal axis, means for maintaining the acid in said reservoir in tangential contact with said cylinder, means for revolving said cylinder at such a speed as to disperse said acid as a fine spray, means for passing ammonia gas through said spray chamber into contact with said spray, and means for discharging acid containing suspended crystals of an ammonium salt from said reservoir, said discharging means positioned near the base of said reservoir so that crystals of the ammonium salt charged into said reservoir by said spray will undergo a substantial growth before being discharged therefrom.

5. An ammonia saturator comprising a relatively high chamber containing a relatively deep body of liquid, means for maintaining a substantially constant level of liquid in said chamber, a spray roll rotatably mounted in the vicinity of the top of said chamber with its surface in substantially tangential contact with the surface of said liquid, a casing enclosing said spray roll and providing a gas space thereabove, said casing having a gas inlet and a gas outlet for introduction and discharge of gas respectively into and from said casing, the gas in its passage through said gas space being intimately contacted with said spray created by the rotation of said roll, and an exit port in the vicinity of the base of said chamber.

6. In an ammonia saturator, the combination which comprises a plurality of cylinders positioned parallel with respect to their longitudinal axes, a spherical shaped spray chamber enclosing said cylinders, said spray chamber having its longitudinal axis transverse to the horizon-

tal axis of said cylinders, an acid reservoir positioned below said spray chamber, means for maintaining a relatively deep body of acid in said reservoir in tangential contact with said cylinders, means for rotating said cylinders at such a speed as to disperse the acid as a fine spray, means for passing a gas through said spray chamber into contact with said spray, and means for discharging a liquid containing suspended crystals from said reservoir, said discharging means positioned in said reservoir at such a point that crystals produced in said reservoir will undergo a substantial growth before being discharged therefrom.

7. In an ammonia saturator, the combination which comprises a reservoir adapted to contain a body of acid liquor through which ammonium salt crystals are adapted to be passed, a spray chamber positioned above said reservoir and arranged to charge a sprayed liquor containing ammonium salt crystals into the liquor contained in said reservoir, and crystal discharge means associated with said reservoir, said discharge means being so positioned with respect to the point of charge of the ammonium salt crystals into said liquor, that crystals passing through said liquor undergo a substantial growth before discharge from said reservoir by said discharging means, and also being positioned to one side of said body of acid liquor to avoid agitating the body of acid liquor and thus interfering with the growth of said crystals.

8. A process for the preparation of salts from the basic and acid constituents thereof, which comprises contacting one of the constituents of a salt in the gaseous phase with a spray of a liquid containing the other constituent of said salt, said spray being of the type obtainable by rapidly rotating a cylindrical surface maintained substantially in tangential contact with a body of said liquid.

9. A process for the preparation of ammonium salts from ammonia and an acid, which comprises contacting ammonia in the gaseous phase with a fine spray of said acid, said spray being of the type obtainable by rapidly rotating a cylindrical surface maintained substantially in tangential contact with a body of said acid.

10. In a process for the preparation of an ammonium salt, the steps which comprise absorbing ammonia in a spray of an acid-containing liquid, introducing the spray containing the absorbed ammonia into an extended body of said liquid without causing substantial agitation of said body of acid-containing liquid, maintaining the products of reaction of the ammonia with the acid in contact with said body of liquid for such a time that crystals produced in said liquid will undergo a substantial increase in size, and withdrawing such large crystals from said extended body of liquid without substantially agitating said body of liquid.

11. In a process for the preparation of ammonium salt, the steps which comprise dispersing a solution of sulfuric acid as a spray, contacting said spray with an ammonia-containing gas whereby crystals of ammonium sulfate are produced in said spray, introducing said spray containing ammonium sulfate crystals into an extended body of said acid saturated with ammonium sulfate, causing the ammonium sulfate crystals to contact therewith for such a time that the crystals will undergo a substantial increase in size and withdrawing such large crystals from the said body of acid without substantially agitating said body of acid.

12. In a process for the preparation of ammonium salt, the steps which comprise maintaining an extended body of an acid-containing liquid, rotating a cylinder in tangential contact with the surface of said liquid maintained at a substantially constant level at such a speed as to disperse said liquid as a fine spray, contacting ammonia gas with said spray, returning the sprayed liquid to said extended body of said liquid, and maintaining the products of reaction of ammonia with the acid in contact with said liquid for such a time that crystals produced in said liquid will undergo a substantial increase in size.

13. In a process for the preparation of ammonium salt, the steps which comprise maintaining an extended body of a liquid containing sulfuric acid, rotating a cylinder in tangential contact with said liquid at such a speed that said liquid will be dispersed as a fine spray, contacting said spray with a stream of an ammonia-containing gas whereby ammonium sulfate is formed, returning the sprayed liquid containing the ammonium sulfate to said body of liquid, maintaining the ammonium sulfate in contact with said liquid for such a time that crystals of said ammonium sulfate produced in said liquid will undergo a substantial increase in size, and separating ammonium sulfate crystals from said liquid.

14. In a process for the preparation of ammonium salt, the steps which comprise maintaining an extended body of an acid-containing liquid, rotating a cylinder in tangential contact with the surface of said liquid at such a speed as to disperse said liquid as a fine spray, contacting ammonia gas with said spray, whereby crystals of an ammonium salt are produced in said spray, charging the sprayed liquid containing said crystals into said body of liquid and discharging said crystals at such a point in said body of liquid that the crystals passing through the body of liquid will undergo a substantial growth before being discharged from said liquid.

15. In a process for the preparation of an ammonium salt, the steps which comprise maintaining a relatively deep body of acid-containing liquid, producing a spray of said acid-containing liquid above said body of liquid without causing substantial agitation of said deep body of liquid, absorbing ammonia in said spray to produce an ammonium salt, causing crystals of said salt thus formed to pass through said deep body of acid-containing liquid to cause the ammonium salt crystals to undergo a substantial increase in size, and withdrawing such large crystals from the body of acid-containing liquid while avoiding substantial agitation of said deep body of acid-containing liquid.

WILLIAM TIDDY.